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**THE SCIENCE MASTERS' BOOK**

**SERIES II**

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**BIOLOGY—CHEMISTRY—  
EXPERIMENTS FOR RECEPTIONS**



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BOOK

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# THE SCIENCE MASTERS' BOOK

SERIES II

## PART II BIOLOGY—CHEMISTRY— EXPERIMENTS FOR RECEPTIONS

BEING EXPERIMENTS SELECTED FROM THE *SCHOOL  
SCIENCE REVIEW* BY A COMMITTEE OF THE SCIENCE  
MASTERS' ASSOCIATION

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## P R E F A C E

THE frequent references to the First Series of the *Science Masters' Book* and the measure of success which it achieved have led the Committee to think that a Second Series may be found acceptable

As in the First Series, many of the notes are original ; on the other hand, several whose names are added to the notes emphatically disclaim original authorship.

It is necessary to repeat that the book is a scrap book, and therefore arrangement in logical sequence without gaps is impossible. It has been thought best to adopt a bald alphabetical classification, supplemented by a displayed list of titles at the beginning and an index to both parts in each volume. It is hoped that this will enable readers to find what they want quickly.

The Committee of the Science Masters' Association offer their sincere thanks to members of the Association, University teachers and others interested in the development of science teaching in schools, who have sent contributions to the *School Science Review* and allowed those contributions to be used again in this collection.



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# BIOCHEMISTRY

## ANALYTICAL

### 1. TESTS FOR ORGANIC FOOD MATERIALS

*H. J. Phelps*

#### *Proteins.*

The tests may conveniently be demonstrated with 0.5 per cent. solutions of gelatin and egg-albumin. Egg-white which has been beaten up with about six times its volume of water will also give albumin reactions quite satisfactorily.

*The Biuret Reaction.*—This reaction is obtained with substances containing two closely adjacent peptide linkages( $\text{—CO—NH—}$ ). Since all proteins are essentially built up from long chains of amino-acids conjugated together by the peptide link, the biuret reaction is a general one for proteins. It is shown also by oxamide, malonamide, many poly-peptides and, of course, biuret itself. The test is carried out by taking in a test-tube not more than one drop of a 1 per cent. solution of copper sulphate and adding to it about 5 c.c. of ordinary bench dilute (about 8 per cent.) caustic soda. This will dissolve the copper hydroxide formed to a solution which should show only the very slightest trace of blue colour. It is most important not to use too much copper sulphate. Add 2 or 3 c.c. of the alkaline copper solution to a roughly equal volume of the solution to be tested. A violet, or pink, colour is formed immediately without heating. The colour may be so weak that it must be observed by looking down the length of the test-tube against a white background, and one is tempted to use a stronger copper



*Sulphur in Proteins.*—Many proteins contain sulphur or phosphorus or both, and it is generally useful, once the protein nature of a substance has been established, to proceed with the tests for these elements. The sulphur test may be illustrated by boiling 1 or 2 c.c. of undiluted egg-white with about  $\frac{1}{2}$  c.c. of 40 per cent. caustic soda for a minute or so. On adding a few drops of lead acetate, the solution turns black and a black precipitate of lead sulphide shortly begins to settle. Most of the sulphur in a protein is in the form of the thio-amino-acid cystein, or the corresponding di-sulphide cystine.

*Phosphorus in Proteins.*—Phosphorus is best tested for by mixing a small quantity of dried protein with at least twice its bulk of fusion mixture (anhydrous  $\text{Na}_2\text{CO}_3$  two parts and  $\text{KNO}_3$  one part) and heating in a crucible at first cautiously until the mass glows, and subsequently strongly until it is wholly fused. Allow the melt to cool and extract it with nitric acid diluted with an equal quantity of water. Proceed to test this solution for inorganic phosphate in the ordinary way with ammonium molybdate, a yellow precipitate being formed on boiling. Casein, which is readily obtained from chemical dealers, gives a strong phosphorus test.

*Insoluble Proteins.*—Although most of the foregoing tests have been described as they are applied to a protein in solution, they may all, with the exception of the biuret test, be obtained with insoluble proteins. They are easily demonstrated with the insoluble protein keratin which is present in finger- and toe-nails. A nail paring will readily develop the characteristic colours of the xanthoprotein, Millon's and glyoxylic reactions, when treated with the appropriate reagents. The colours, of course, develop on the nail paring itself and do not diffuse through the bulk of the solution to any extent. For the glyoxylic reaction to be shown it is necessary to see that the paring comes into contact with both the glyoxylic reagent and the strong sulphuric acid layer beneath it.

*Precipitation of Proteins.*—Protein solutions are pre-

precipitated by salts of heavy metals. This may be illustrated with lead acetate (or basic lead acetate), mercurous nitrate (or Millon's reagent) and with copper sulphate. Albumin is more easily precipitated than gelatin under these conditions. The proteins are also precipitated by the so-called "alkaloidal" reagents, of which ferrocyanic acid (acid potassium ferrocyanide solution), picric acid and tannic acid are good examples. A solution of 4 gm. of tannic acid in 8 c.c. of strong acetic acid and 190 c.c. of 50 per cent. alcohol, known as Almén's reagent, is a very effective precipitant. An alkaloidal reagent which is much used in the clinical examination of urine for albumin is a 20 per cent. solution of sulpho-salicylic acid.

*Heat Coagulation of Proteins.*—In common with other amphoteric electrolytes, the proteins are much less soluble at the iso-electric point than under any other conditions. If then a solution of a protein is brought to its iso-electric point, a considerable precipitate is formed. This precipitate is soluble in excess acid or alkali in the cold, but if it is thrown down from hot solution or if it is boiled after formation, it is rapidly "coagulated" and converted to an insoluble form. This phenomenon may be illustrated with a solution of albumin. First, add dilute acetic acid drop by drop and note the formation of a precipitate which is easily soluble in excess acid. Now take a sample of albumin solution heated almost to boiling-point and add very dilute acetic acid carefully until no more precipitate is formed. Next boil the solution for a short time and it will be seen that the precipitate rapidly flocculates and settles, leaving the solution almost water-clear. The precipitate so formed will not dissolve in excess acid. It is important to note that gelatin does not show the phenomenon of "heat coagulation"; it may, however, be removed from solution by half-saturation with ammonium sulphate (a state of affairs most easily attained by adding to a protein solution an equal volume of a saturated solution of ammonium sulphate in

water). All proteins are insoluble in *fully* saturated ammonium sulphate.

### *Carbohydrates.*

*Benedict's Reduction Test.*—In the absence of protein, Molisch's reaction may be used to establish the presence of some form of carbohydrate. It is next desirable to apply a series of reduction tests. The familiar *Fehling's* test needs no description; certain other tests are found very valuable also in biochemical work and some details of the necessary reagents may not be out of place. *Benedict's* reduction test will be found on the whole more valuable than *Fehling's* since *Benedict's* copper reagent is not so readily reduced by uric acid, chloroform, or creatinine. *Benedict's* solution is best prepared as follows. Dissolve 173 gm. of sodium citrate and 90 gm. of anhydrous sodium carbonate in about 600 c.c. of hot distilled water; filter and make up to 850 c.c. Dissolve 17.3 gm. of copper sulphate in about 100 c.c. of water and make up to 150 c.c. Add the copper solution slowly, with constant stirring, to the carbonate-citrate solution. The resulting mixture is ready for use and keeps well. All reducing sugars readily reduce *Benedict's* solution at the boiling-point. As with *Fehling's* solution, the colour of the precipitated cuprous oxide depends greatly on the concentration of the reducing sugar. In general, it is as well to use *Benedict's* test in addition to, and not instead of, *Fehling's* test.

*Distinction between Mono- and di-saccharides. Barfoed's Test.*—If the presence of reducing sugar is established by one of the above tests, *Barfoed's* test should be applied to distinguish between mono-saccharides and reducing di-saccharides. *Barfoed's* test requires an acid solution of copper acetate, which is prepared by dissolving 45 gm. of neutral copper acetate in 900 c.c. of water, filtering and adding 12 c.c. of 50 per cent. acetic acid to the filtrate, and making the total volume up to one litre. This reagent is in fact reduced by almost all sugars if it is boiled

with them in sufficient concentration for a sufficient time. It is, however, much more easily reduced by mono-saccharides than by di-saccharides. This may be illustrated by heating 1 c.c. of a 0.2 per cent. glucose solution with about 5 c.c. of Barfoed's reagent in a boiling water-bath for three or four minutes, when a strong reduction is obtained. Repeat the test, using a much stronger maltose solution, say 1 per cent.; very little or no reduction will be observed, although 1 per cent. maltose reduces Fehling's and Benedict's reagents very strongly. In general, it may be assumed that any carbohydrate which reduces alkaline copper reagents strongly and Barfoed's reagent very weakly is a di-saccharide.

*Stability to Alkali.*—Mono-saccharides may also be distinguished from di-saccharides by the relatively greater rapidity with which the mono-saccharides are destroyed by strong alkalis. If 2 c.c. of 1 per cent. glucose are boiled for about one and a half minutes with an equal volume of 40 per cent. caustic soda, no reduction can subsequently be obtained. If maltose is similarly treated, the reducing power is not entirely destroyed.

*Precipitation of Starch.*—The presence of starch in biological material is demonstrated by the formation of the familiar blue colour with iodine,<sup>1</sup> and is often to be inferred from its insolubility in cold water and the formation of opalescent solutions on boiling, since naturally occurring starch is normally in the granular form. The colloidal solutions produced by boiling granular starch in water are completely precipitated by half-saturation with ammonium sulphate. It should be pointed out, however, that solutions of "soluble" starch are only very slowly precipitated by this treatment, although full saturation with ammonium sulphate rapidly precipitates all forms of starch. The primary products of the hydrolysis of starch, the dextrans, are frequently found in associa-

<sup>1</sup> It is better to boil solutions before applying the iodine test for starch. Native starch granules give an indefinite greenish-black colour.

tion with it. In this case starch must be precipitated with ammonium sulphate before the characteristic dextrin reactions can be demonstrated. There are two main types of dextrans—the erythro-dextrans which give a red colour with iodine and the achroo-dextrans which give no colour with iodine. It must be noted that in practice it is very difficult to get dextrin entirely free from starch, and the first effect of the gradual addition of dilute iodine to a dextrin solution is almost always the formation of a blue colour, which is rapidly masked by the developing red colour if dextrin is present.

*Hydrolysis of Starch.*—The successive stages in the hydrolysis of starch through the dextrans and maltose to glucose may be illustrated by two simple experiments. First, take a 1 per cent. solution of “soluble” starch and to 25 c.c. of this add 1 c.c. of strong hydrochloric acid, mixing very thoroughly. Divide the solution into five equal portions and place four of them at the same instant in a boiling water-bath. Remove the tubes and cool them quickly after intervals of three, six, nine and twelve minutes. Divide the contents of each tube into two equal portions and test one with iodine and the other with Fehling’s solution. It will be seen that the iodine reaction is successively blue, purple, red, and colourless, while the reducing power gradually increases, usually first becoming noticeable after about six minutes’ heating.

*Enzymic Hydrolysis of Starch.*—The enzymic hydrolysis of starch may be illustrated by a similar experiment. A solution of ptyalin, the starch-splitting enzyme of the saliva, can be obtained by washing the mouth out with a small quantity of water. Place 3 c.c. of a 1 per cent. suspension of granular starch in each of five tubes and add to each tube an equal volume of diluted saliva. Place the tubes in a water-bath kept at 40° C., and after various intervals, remove the tubes and test with iodine and with Fehling’s solution. The concentration of ptyalin obtained in saliva under these conditions is so variable that it is impossible to suggest exact times of heating, but

if the first tube is examined after two or three minutes, it should be possible, by estimating the amount of hydrolysis that has taken place in that time, to arrange a timetable for the remaining tubes. It should be pointed out that the action of ptyalin only hydrolyses starch to maltose, the subsequent breakdown of maltose to glucose in the animal depending on a specific enzyme.

*Test for Sucrose*—The familiar non-reducing di-saccharide sucrose does not give any of the tests so far mentioned. Its presence is easily recognized, however, by hydrolysing it to give glucose and fructose. If about 3 c.c. of cane sugar solution are boiled for a few seconds with one or two drops of strong hydrochloric acid, the solution, after cooling and neutralizing, shows marked reducing properties. The presence of cane sugar and also of fructose may be confirmed by a specific colour test for fructose, the *Seliwanoff* reaction. This test requires the preparation of a special reagent by dissolving 0.05 gm. of resorcin in 100 c.c. of hydrochloric acid (made by diluting the strong acid with its own volume of water). If 5 c.c. of this reagent are warmed with a few drops of fructose solution an intense red colour is produced, and often a red precipitate is also formed. Cane sugar under these conditions is immediately hydrolysed by the hydrochloric acid present and gives the fructose reaction. Glucose gives *Seliwanoff's* reaction to a slight extent after very long boiling, but the colour is never as intense as fructose and a red precipitate is never formed.

*Preparation of Osazones*.—All reducing sugars may be characterized by the crystalline forms of their osazones. The general method for preparing osazones is as follows. To about 10 c.c. of the solution to be tested, add about 1 c.c. of strong acetic acid. Now add as much solid phenyl-hydrazine hydrochloride as will lie on a sixpence, and at least twice as much solid sodium acetate. Warm the tube until these substances are dissolved and filter. Place the tube in a boiling water-bath for about half an hour. Glucosazone, if present, will probably appear as

a yellow precipitate, while the solution is still hot, but the osazones of maltose, lactose and other reducing sugars will not be precipitated until cool. Finally, it might be pointed out that the common sugars, glucose and fructose, form the same osazone, and particularly it should be emphasized that only reducing sugars form osazones.

### *Fats.*

A property of fat which is of the greatest importance from the biological point of view is the formation of stable emulsions in alkaline solutions. This is easily shown by shaking olive oil with water to which one drop of dilute caustic soda has been added. The emulsion formed is stabilized by the formation of a small quantity of sodium oleate.

*Osmic Acid Test.*—Certain characteristic reactions of the fats depend on the reducing power of unsaturated organic compounds. If a very small amount of a fat is treated with a few drops of 1 per cent. osmic acid solution, the fat is quickly blackened. The solid fats contain enough unsaturated fatty acids to give this reaction. Osmic acid may be purchased in 1 per cent. solution. The degree of unsaturation of a fat is determined quantitatively by measuring the amount of iodine that is taken up by a given weight of fat from a solution of iodine and iodine trichloride in glacial acetic acid.

*Hydrolysis of Fats: Production of Glycerol.*—The glyceride nature of fats is easily shown by hydrolysis. If about 5 gm. of lard are boiled for twenty minutes or so with 25 c.c. of alcoholic potash, the fat is hydrolysed to glycerol and the potassium salts of palmitic and stearic acids (potassium soaps). If the hydrolysed liquid is diluted with an equal volume of water and the alcohol driven off by heating on a water-bath, the free fatty acids may be precipitated by acidifying with sulphuric acid and filtered off. The presence of glycerol in the filtrate is demonstrated by first neutralizing and evaporating to a syrup, from which the sodium sulphate present

is removed by adding alcohol. The alcoholic solution is then evaporated as far as possible on the water-bath and tested for glycerol by heating with dry acid potassium sulphate, when the characteristic smell of acrolein will be detected.

## 2. SIMPLE ENZYME REACTIONS

*H. J. Phelps*

*Preparation of Fibrin.*—Obtain some *fresh* blood from a slaughter-house, and whip it with a wire whisk for sufficient time for the fibrin to collect on the whisk. Remove the fibrin and wash free from blood.

(Several wire pipe-cleaners make a satisfactory whisk.—E. J. M.)

*Preparation of “ Congo Fibrin.”*—For certain experiments on the action of proteolytic enzymes, fibrin dyed with Congo Red is necessary. “ Congo Fibrin ” is prepared as follows :

Fibrin, prepared and washed as already described, is soaked for twenty-four hours in a 0.01 per cent. solution of congo red, in the proportions of about 50 gm. of wet fibrin per 100 c.c. of solution. The fibrin and dye solution are then poured into excess of water and heated to about 80° C. for twenty minutes. The fibrin is then collected on a cloth and washed under the tap for some time. It should be squeezed as dry as possible and kept in a mixture of equal parts of glycerol and water, to which a little toluol is added, to prevent the growth of moulds. The digestion of such fibrin can easily be followed, since the congo red is liberated as the protein is dissolved.

*Comparison of the Proteolytic Action of Pepsin and Trypsin on Fibrin.*—In the following experiment, use commercial pepsin solution diluted with two parts of distilled water, and commercial trypsin solution at full strength.



With each enzyme set up two series of four test-tubes, to each of which is added a small piece of fibrin, all the pieces of fibrin being approximately the same size. To the first four tubes add respectively (a) 1 c.c. of diluted pepsin and 1 c.c. of dilute (about 2N) hydrochloric acid; (b) 1 c.c. of pepsin and 1 c.c. of dilute sodium carbonate; (c) 1 c.c. of water and 1 c.c. of dilute hydrochloric acid; (d) 1 c.c. of pepsin and 1 c.c. of dilute hydrochloric acid, and boil the whole at once for a few moments. To the remaining tubes add respectively, (a) 1 c.c. of trypsin solution and 1 c.c. of hydrochloric acid; (b) 1 c.c. of trypsin and 1 c.c. of sodium carbonate solution; (c) 1 c.c. of water and 1 c.c. of sodium carbonate solution; (d) 1 c.c. of trypsin and 1 c.c. of sodium carbonate, and boil the whole for a few moments.

Cool the tubes which have been boiled and place all the tubes in a water-bath at about 40° C. It will be noted that in the presence of acid congo red gives a dark blue colour. For comparative purposes, it will therefore be necessary to neutralize the acid tubes with a little strong caustic soda at the conclusion of the experiment. When the experiment has been in progress long enough for a considerable amount of dissolved congo red to have appeared in trypsin tube (b), remove all the tubes from the water-bath and neutralize the acid tubes. The amount of congo red in solution will then give an approximate idea of the activity of the two enzymes under the conditions of the experiments.

It will easily be seen that pepsin is active in acid solutions and that trypsin is active in alkaline solutions, and that boiling inactivates both enzymes. The control experiments show that neither hydrochloric acid nor sodium carbonate by itself has any action on the fibrin.

*Inactivation of Pepsin by Alkali.*—Take pepsin tube "b" of the previous experiment and make it acid to litmus by adding dilute hydrochloric acid. Replace the tube in the water-bath at 40° C. and leave for some time.

It will be found that there is no action on the fibrin, indicating that the *previous* heating with sodium carbonate has inactivated the pepsin.

*Clotting of Milk.*—(a) Heat 1 c.c. of commercial pepsin and 5 c.c. of milk in a test-tube in a water-bath at  $38^{\circ}$ – $40^{\circ}$  C. for a short time. The milk will quickly curdle, and if left in the water-bath, a clot will contract, leaving a clear fluid.

(b) Repeat the experiment, using commercial rennin in place of pepsin, when a similar clot-formation will occur.

*To show that the Clotting of Milk depends on the Presence of Calcium*—Remove the calcium from a sample of milk by adding to 100 c.c. of milk about 30 c.c. of N/5 ammonium oxalate. With this “decalcified” milk set up a series of three tubes, containing in the first 3 c.c. of oxalated milk together with 1 c.c. of N calcium chloride solution and 1 c.c. of commercial rennin preparation; in the second 3 c.c. of milk and 1 c.c. of rennin; to the third 3 c.c. of milk and 1 c.c. of rennin preparation which has been boiled. Place all three tubes in a water bath at  $40^{\circ}$  C. for at least ten minutes, or until a clot has formed in the first tube. Next, boil the contents of the second tube for a short time to destroy the rennin. After cooling this tube, add about 1 c.c. of calcium chloride solution to both the second and third tubes. The contents of the second tube clot at once, thus demonstrating that the hydrolysis of casein by rennin, which is the essential preliminary of clot formation, has proceeded normally in the absence of calcium, although no clot will form until calcium is supplied. The contents of the third tube do not form a clot since the rennin is inactivated by the preliminary boiling.

*To Show that the Action of Trypsin varies with the Hydrogen-ion Concentration of the Medium.*—A one-twentieth molar solution of sodium borate and a one-fifth molar solution of boric acid, to which is added enough sodium chloride to make the solution one-twentieth molar

in that salt, are required. With these solutions make up a series of eight 10-c.c. samples as follows :

Tube.	Sodium Borate Solution.	Boric Acid Solution.	pH of Mixture
A . . . . .	1.0 c.c.	9.0 c.c.	7.4
B . . . . .	2.0 "	8.0 "	7.8
C . . . . .	3.0 "	7.0 "	8.1
D . . . . .	4.0 "	6.0 "	8.3
E . . . . .	5.0 "	5.0 "	8.5
F . . . . .	6.0 "	4.0 "	8.7
G . . . . .	8.0 "	2.0 "	9.0
H . . . . .	10.0 "	nil	9.2

In each tube place one of eight equal-sized pieces of fibrin. Add to each tube 1 c.c. of commercial trypsin and place the tubes in a water-bath at 40° C. for about half an hour. At the end of this period, it will probably be obvious that more congo red has been dissolved in tubes C and D than in the others. If a rough colorimeter is available, it is possible by comparing the actual intensities of congo red in the various tubes to get a roughly quantitative measure of the activity of trypsin over the range of reaction studied. [It should be noted that, in the author's experience, some samples of commercial trypsin show a second "optimum" in the most alkaline tubes, owing to the presence of some impurity.]

*A Rough Estimation of the Activity of a Sample of Pepsin.*  
—Prepare "calcified" milk by adding 10 c.c. of a normal solution of calcium chloride to 50 c.c. of milk and making the total volume up to 100 c.c. In each of five test-tubes place 5 c.c. of "calcified" milk and warm all the tubes to 40° C. in a water-bath. To one tube add 1 c.c. of the pepsin solution under test, mix quickly and replace in the bath. Examine the tube from time to time and note when a definite clot first forms. If the clotting time is very short, dilute the enzyme solution five or ten times and repeat the test. Discover in this way a dilution of enzyme which gives clotting in something between one and a half and one and three-quarter minutes. Measure this time as accurately as possible. The unit suggested by Cole (*Practical Physiological Chemistry*) is that amount of

enzyme which will clot 5 c.c. of calcified milk in 100 seconds. From the dilutions which have been made in the test and the final clotting time, the strength of the original enzyme solution may be expressed in terms of this unit.

*Clotting of Blood.*—Add absolutely fresh slaughter-house blood, or blood from a newly-killed animal, to 1 per cent. potassium oxalate in the proportion of 1 litre of blood to 100 c.c. of oxalate solution. If a centrifuge is available, the formation of the clot can be more exactly studied by first removing the red blood cells by centrifugation and pipetting off the supernatant plasma. In this way also the formation of the clot is shown to be independent of the presence of red cells. Dilute about 5 c.c. of the oxalated plasma, so obtained, with about twice its volume of water and divide it into two portions. To one add about 0.5 c.c. of 1 per cent. calcium chloride solution. Place both tubes in a water bath at 40° C. The plasma to which calcium chloride has been added will rapidly form a clot, which, in the absence of red cells, will be an almost clear jelly.

*Inhibition of the Clotting of Blood by High Concentrations of Salts.*—Add fresh blood to saturated magnesium sulphate in the proportion of 1 litre of blood to 200 c.c. of magnesium sulphate, and, if possible, removing the red cells by centrifugation, as before. Such “salted plasma” will not clot. If, however, a sample of it is diluted with about five times its volume of water and kept at 40° C., a clot slowly forms, since the magnesium sulphate has become so diluted that any fibrin formed is now insoluble and is precipitated in the normal way.

*Demonstration of an Oxidase in the Potato Tuber.*—Prepare an alcoholic solution of guaiacum resin by heating 15 gm. of fresh broken resin in 100 c.c. of 95 per cent. alcohol in a flask, containing a little absorbent charcoal, on a water-bath for five minutes and then filtering.

Crush up a small raw potato in water and filter off the solid residue. If guaiacum be added to the filtrate, a blue

colour is soon developed. If, however, a sample of the filtrate be kept at 65° C. for a few minutes, it will be found that a blue colour is only very slowly formed; if hydrogen peroxide be added, however, the solution rapidly turns blue. The probable explanation of this is that heating to 65° C. has largely destroyed the organic peroxide or its precursor, but has not inactivated the peroxidase.

*Demonstration of a Peroxidase in Horse-Radish Root.*—Pound some horse-radish scrapings in alcohol and filter off the solid residue. Evaporate the filtrate to dryness, and take up the solid obtained in water; filter if necessary. With the peroxidase extract so obtained, set up a series of three tubes; one with 3 c.c. of the extract and a little guaiacum; another with 3 c.c. of the extract and a little guaiacum, together with a few drops of hydrogen peroxide, and a third tube with guaiacum tincture and hydrogen peroxide only. A blue colour develops only in the tube which contains the peroxidase extract together with guaiacum and hydrogen peroxide.

*The "Benzidine Reaction" of Blood.*<sup>1</sup>—Prepare a fresh solution of benzidine in acetic acid. To the benzidine solution add an equal volume of hydrogen peroxide and 1 c.c. of a very dilute solution of blood—say 1 drop of blood in 100 c.c. of water—an intense blue colour develops. It is interesting to discover the maximum dilution of blood which will give this test.

*Demonstration of Urease in Soya Bean.*—Prepare a dilute solution of urea and add a few drops of phenol red; next add dilute acetic acid until the acid (yellow) colour of phenol red is shown. Add some soya bean meal and heat the mixture to 45° C. The solution will soon develop

<sup>1</sup> This is *not* a specific test for blood. The blue reaction is not necessarily due to peroxidase, though it may be produced under various conditions by a number of organic materials. As the reaction is given by blood which has been boiled, and in which, presumably, any enzyme systems have been destroyed, these cannot be the active factor in this case. In blood, hæmoglobin causes the reaction, but its iron-containing derivatives are also active: iodides and various other salts can produce the colour. The value of the test lies in the fact that, if negative, blood cannot be suspected, but, if positive, that it may be worth while to apply more conclusive tests for its presence.—E. J. M.

the alkaline (red-purple) colour of the indicator as alkaline ammonium carbonate is formed by the decomposition of the urea.

## CHLOROPHYLL

### 3. EXPERIMENTS WITH CHLOROPHYLL

*James Gillespie*

*Fluorescence of Chlorophyll.*—The ability to absorb light of a certain wavelength and to emit light of a different wave-length can very easily be demonstrated with a solution of chlorophyll in ether or acetone. Viewed by direct light, the solution has a pronounced green colour, but with transmitted light the solution appears to be blood-red. For a quick demonstration of this phenomenon, the most convenient method is to suspend a flask of chlorophyll solution between an electric lamp and a mirror hung parallel to the latter. When one looks into the flask, only a green-coloured fluid is seen, but the image in the mirror is of a deep red colour. Even without this apparatus, if the observer merely alters the angle at which he is viewing the solution, he can see a change-over from the typical green to the red; standing the flask containing the solution on a mirror is of great assistance in this connection. *Experiments in fluorescence should always be done with freshly-prepared solutions because the property of emitting red rays wears off as the solution ages.*

*Extraction of the Chloroplast Pigments.*—Possibly the most favourable material for extraction experiments is the leaves of the common Stinging Nettle (*Urtica dioica*). These can be dried slowly in air, powdered finely and kept indefinitely, so that extraction can be carried out at any season. The obvious advantage of powdered material is that work can be done with small bulks. The writer can recommend the dried leaves of Wallflower and Red Currant, but Elder and Conifer leaves are spoilt by drying. The four pigments in the plastid

are soluble in 80 per cent acetone, and in this connection a point of interest and importance should be noted. Completely dry leaf powder, when treated with anhydrous acetone, fails to yield anything more than a faint trace of greenness, but on the addition of even a few drops of water, the pigments become readily soluble.

For demonstration in class, 3–4 gm. of leaf powder will give a deep green solution. The material should be spread out on a filter paper in a Büchner funnel, and 10 c.c. of the 80 per cent. acetone allowed to soak into it thoroughly, the fluid is then sucked through into the flask and the operation repeated.

*Demonstration of Chlorophylls a and b.*—Add the acetone extract to double the amount of petroleum ether in a separating funnel, and remove all traces of acetone in the following manner. Very carefully run an equal volume of distilled water down the side of the funnel (if this operation is not done with care an emulsion will be formed, making operation very difficult or impossible). The ethereal layer containing the pigments will separate out on top, leaving a lower greenish watery layer, which is then run off. The process should be repeated three or four times. Now take a few c.c. of the petroleum ether solution and shake with an equal amount of 92 per cent. methyl alcohol. Two layers separate out, the uppermost being an ethereal solution of chlorophyll *a* plus carotin, and the lower a methyl alcoholic solution of chlorophyll *b* with xanthophyll. The carotin and the xanthophyll obscure to some extent the difference in colour between the two layers; but despite this, one can still see the blue-green of the chlorophyll *a* and the purer green of the chlorophyll *b*.

*Separation of Chlorophylls a and b from Carotin and Xanthophyll*—About 5 c.c. of the petroleum ether solution of the "crude" chlorophyll are shaken with roughly 2 c.c. of concentrated methyl alcoholic potash. The green colour disappears, but reappears slowly. When the solution is fully green, slowly add 10 c.c. of water and

then a little more ether. Vigorous shaking causes the separation of the two layers, an upper ethereal layer of the carotinoids and a lower watery alkaline solution of chlorophyllin.

*Separation of the Two Carotinoids.*—Add the ethereal solution of the carotin and the xanthophyll to a separating

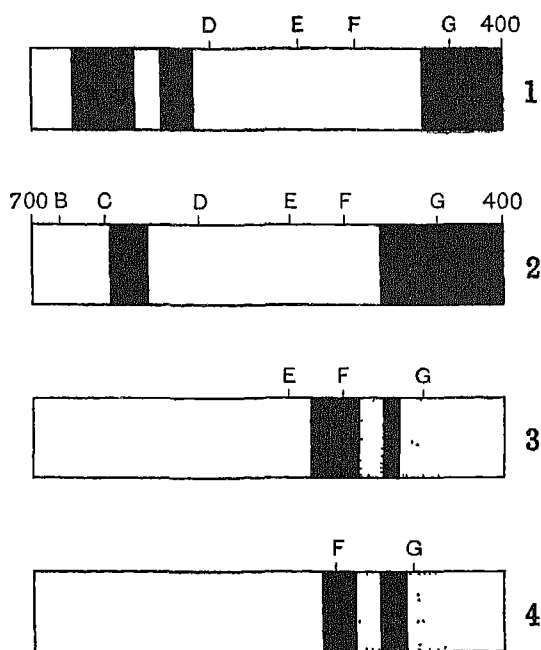


FIG. 1.—Absorption Spectra of the Chloroplast Pigments  
 1.—Chlorophyll *a*. 2 —Chlorophyll *b*. 3.—Carotin.  
 4 —Xanthophyll.

Only the main bands are shown.

funnel, gently pour in a little water and then run it off again. Repeat until the ether solution is thoroughly washed. Transfer it to an evaporating basin and concentrate to about 1 c.c. Now add 10 c.c. of light petroleum and shake with 10 c.c. of 90 per cent. methyl alcohol until the latter shows no colour. The light petroleum contains



the carotin, while the xanthophyll is in the methyl alcohol.

The complete separation of chlorophyll *a* from chlorophyll *b* involves more difficulty than any of the above operations, and is usually too troublesome to do satisfactorily in class.

*Spectroscopic Examination of Chlorophyll.*—The green pigments of the chlorophyll complex yield interesting spectroscopic data concerning their relation to light. For this study, an ordinary hand spectroscope will serve admirably, and its value is, of course, much greater if it possesses a scale of wave-lengths superimposed on the spectrum band. The complex is extracted from the leaves as described, and the solution of the pigments in acetone is added to a glass container with flat sides. The spectroscope is calibrated against a sodium flame to determine the exact position of the D Fraunhofer line, and then directed towards a strong source of light, the slit of the front being brought close to the sides of the glass container. Only some of the absorption bands of the chlorophyll complex are visible if a concentrated solution is used; but whether dilute or concentrated, the solution will show a broad absorption band in the red end of the spectrum, a weaker band in the orange and a second broad band in the blue-violet. Fig. 1 shows typical spectra for all four pigments.

## BIOLOGY

### GENERAL MAINTENANCE

#### 4. STEAM STERILIZER

*L. W. White*

Obtain a large biscuit tin (1s. from any grocer) and solder all the seams, so as to make sure it is watertight. It is better to solder inside the tin than outside and I have

found zinc chloride solution better than a paste flux. Cut four pieces of asbestos millboard to fit the sides of the tin (outside) and secure them in position with wire. A cover in the form of a pyramid is required, so that a large flask will fit inside the sterilizer.

This cover should be cut from tin plate in one piece (an interesting exercise in practical geometry <sup>1</sup>) and then folded, the seam soldered, and a small rectangle of tin plate soldered on the top. With a pair of pliers, bend the edges at the base so that the cover fits the tin well. Four beehive shelves placed inside the tin make a convenient platform on which to stand flasks, beakers, etc. If about 3 in. of water is placed in the sterilizer, it may be kept gently boiling for three or four hours without replenishment.

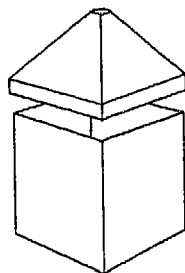


FIG. 2.

## 5 CROSS-WIRE EYEPiece

*Eric Ashby*

The following simple method of making a cross-wire

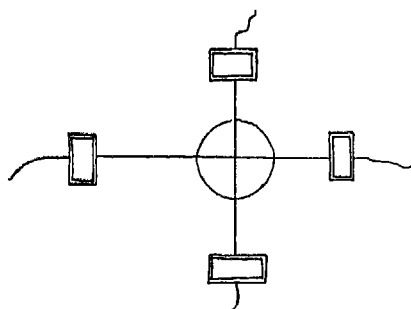


FIG. 3.

eyepiece for the microscope was taught by the late Professor Chodat, at Geneva. Lay two hairs, taken from a student whose hair is not oiled, at right angles on a piece of paper. Stretch them as tightly as possible, and stick down their

ends to the paper with gum labels. Slide a  $\frac{3}{4}$ -in. cover-slip under the intersection of the hairs (Fig. 3). Put a drop of fairly stiff warm Canada Balsam on the cover-

slip, over the hairs at their point of intersection, and lay another cover-slip over the top. The intersecting hairs are now set in Canada Balsam between the cover-slips. Do not cut the hairs until the balsam is quite dry and set ; then sever them with a sharp scalpel.

Unscrew the top of the eyepiece and drop the cross-wire on to the stage of the eyepiece. The field of view is now divided into four quadrants, and counting or drawing is greatly facilitated.

## 6. DISSECTING TRAY

*B. M. Denham*

The usual arrangement for dissecting under water is a dish half-filled with paraffin wax on to which the frog, worm, etc., is fastened by pins thrust in at suitable angles. The wax is held down by projections soldered inside the dish or, in the case of enamelled dishes, by loading the wax with lead shot. The following arrangement, which requires no wax, has been found to be cheap and efficient. It has the advantages that there is no obstruction by projecting pins, and the dish can be cleaned more thoroughly after use.

A rectangular piece of perforated zinc of suitable size (say 10 in.  $\times$  8 in.) is obtained. The type required is sold by ironmongers for ventilators, etc. A square is snipped out of each corner of about  $\frac{1}{2}$ -in. side. Each edge is then bent down at right-angles to form a support for the central rectangle. This completes the tray, which is placed in the dish of water.

To fix the object to the tray, it is necessary to make hooks of special shape. With a small pair of pliers and a supply of pins this is the work of a few minutes. About  $\frac{1}{8}$ -in. of the pointed end of the pin is bent right over to form a hook. About  $\frac{1}{4}$ -in. of the head end is bent over through rather more than a right-angle in the direction of the point. The object can now be hooked at suit-

able places and the heads of the pins pushed into appropriate holes, where they will remain fixed securely until released.

## 7. EXPERIMENTAL TABLE FOR ANIMAL PHYSIOLOGY

*G. N. Ridley*

The table was designed to form a compact portable unit for work on the Frog. (See "Physiology of the Heart," *School Science Review* [53], Oct., 1932, p. 68, and "Physiology of Vol. Muscle," *School Science Review* [57], Oct., 1933, p. 80). The arrangement of the component parts is shown in the diagrams.

*Lever pillar* : made of glass rods inserted in large corks

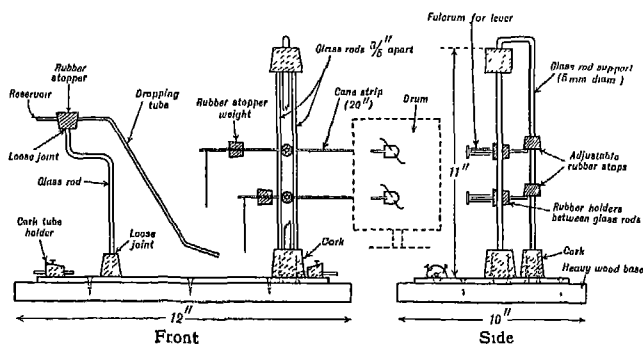


FIG. 4

(1.5 m. diam.). The base corks are glued to the surface of the table and strengthened with wire nails.

*Fulcrum* : 3 mm. diam glass rod held in a rubber stopper of such a size that it grips firmly when pushed between the glass uprights. The stopper is prevented from twisting vertically by a sliding rubber stop on the back upright, moved down to meet the distal end of the fulcrum rod

*Lever rod* : a 20-in. cane (sold for gardening purposes)

split into two. Each strip is planed down to make it as light as possible. About 4.5 in. from one end, a short piece of glass tube is cemented at right-angles to the flat side, this slips over the fulcrum rod. (The writing point is of fine glass held in a paper tab.)

*Dropping tube* : convenient for keeping exposed organs wet with saline during the course of an experiment. The tube and support are both freely movable, enabling the jet to be swung into any desired position over the preparation. The tube is connected to a bottle of the fluid.

*Cork holders* : enable any tubes connected to the preparation to be pinned down to the cork table.

*Cork table* . an ordinary table mat, 11 in.  $\times$  8 in. When the permanent structures have been mounted, the mat is screwed down to a heavy wood base. The cork surface is given a thin coating of paraffin wax.

A valuable addition to the apparatus is a stimulus marker. A sixpenny bell part—electro-magnet and striker—is connected up and operated by a push-button switch. A wire extension is added to the arm of the striker, and a writing point on the free end is allowed to inscribe a continuous line on the drum at a convenient distance below the tracings during the experiment. The moment of application of any stimulus is marked by pressing the switch so that the striker “pen” records a vertical stroke as the arm is pulled up to the magnet.

## 8. ACCESSORY FOR THE EXPERIMENTAL TABLE

*G. N. Ridley*

This simple unit is pinned down in position on the cork table shown in the previous note. It is intended for use in experiments with isolated tissues of the Frog, e.g. ring of stomach or intestine, bladder, muscle-nerve, which are required to be kept immersed in aerated saline solutions. Small *glass* hooks should be used for stomach and

intestine preparations. Chemicals and gland extracts whose physiological actions are to be tested are added direct to the immersion tube (a small specimen tube with the bottom removed), which is drained when necessary through the right-hand tube and refilled from a stock

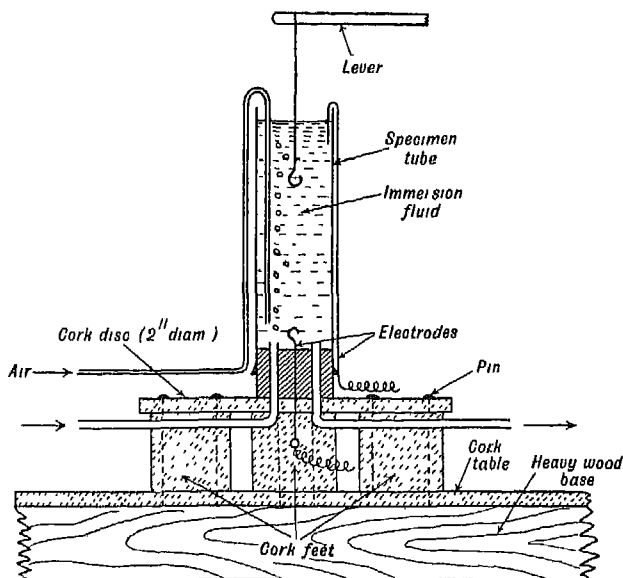


FIG. 5.

bottle through the left. Provision is made for the application of electrical stimuli by means of the pin which passes right through the rubber stopper, one end being hooked, the other being connected to the source of stimulus (cell or induction coil).

## 9 BIOLOGICAL MODELS

*Rev R. Simpson*

*Models of Protozoa in Gelatine.*—For models of protozoa a special colourless gelatine is the most suitable medium. This is prepared by soaking the best commercial gelatine in several changes of cold water and then melting on a

water-bath in the smallest quantity of distilled water. If the melting be done carefully, no charring or discoloration is produced. A quantity of glycerine jelly is then added, together with a little chloroform or menthol. This mass, on cooling, will form a colourless jelly of a suitable consistency. Approximate proportions: gelatine, 75 per cent.; glycerine jelly, 25 per cent.

The mould may be prepared from dentist's modelling wax or plaster of Paris; the former possesses the advantages of being easier to use, less troublesome to prepare and readily melted down for other models. A smooth cake of wax is obtained and the outline of the animal marked. Then with a knife or gouge, the wax is dug out and when a sufficient depth has been obtained, the depression is smoothed with fine glass paper. The hollow is then well vaselined and the molten gelatine poured in. Before setting has proceeded too far the nucleus, food particles, chromatophores, etc., are placed in position as required. A suitable nucleus may be made from a small ball of "Glitter-wax" (obtainable in boxes containing various colours, from most toy shops); while excellent chromatophores may be produced with the common Duckweed. The contractile vacuoles require care, but with a little practice a bubble may be blown in the gelatine in the required position.

When the gelatine has solidified, the model is readily removed from the mould and, after trimming with a warm section lifter, may be mounted on a glass plate in a glass cell containing formalin. When illuminated from below, the structures stand out distinctly and convey a very clear impression of the animal. Large-scale models of *Amoeba*, *Euglena*, *Paramecium* and several other protozoa can be prepared in this way, besides a series of models illustrating the processes of mitotic division.

*Larger Models in Glitter-Wax.*—For larger models, glitter-wax forms an excellent material, and it has been used to prepare models such as the following:

- (i) Sectional view of frog's heart.
- (ii) Circulation of the earthworm.
- (iii) The mammalian ear.
- (iv) Brain and cranial nerves of dogfish.
- (v) Life history of malarial parasite (*Plasmodium vivax*).

Of these the first and third were prepared by carving out the wax ; the second and fourth by covering a wire skeleton with wax of various colours. The models may be erected on wooden bases, in the case of the larger ones, or mounted on cards and framed in the case of the smaller, such as the fifth.

In some cases, e.g. in preparing a model of a sea-anemone or hydra, the outer wall is best prepared from dentist's modelling wax and subsequently covering this with "Luc" cellulose lacquer of the required colour.

The models so prepared will well repay the time expended upon them and are of infinitely greater value than mere diagrams can ever hope to be.

*Circulatory System of an Earthworm.*—A model of the circulatory system of an earthworm is prepared as follows. The framework consists of non-insulated copper wire of about 18 S.W.G. attached by wooden uprights to a wooden base. Three main strands represent the dorsal, sub-intestinal and sub-neural vessels. At the anterior end the dorsal strand is connected to a number of thinner wires which represent the plexus of vessels in the region of segments 1-3. Ventrally these wires are attached to the sub-neural strand, and a similar arrangement prevails at the posterior end. On the dorsal vessel the hearts, constructed of thicker wire, are attached to the sub-intestinal vessel. The parietal vessels are attached as hoops of rather thin wire (20 S.W.G.) between the dorsal and sub-neural vessels, and the afferent nephridial and branchial vessels of still thinner wire (22 S.W.G.) are fixed to the ventral vessel at one end and to the wooden base at the other, their distribution being indicated by means of labels. The efferent nephridial and branchial vessels



are similarly indicated by means of labels on the base of the model and thin wires attached to the parietals.

When the whole framework is constructed, the surface of the wires may be roughened slightly by means of glass paper so that it may hold the wax covering, and glitter-wax (softened by immersion in hot water) is wrapped round as evenly as possible. The colours should vary for the different vessels to indicate the nature of the contained blood, and the plexuses at the anterior and posterior ends should be coloured dorsally as for the dorsal vessel and ventrally as for the sub-neural vessel. The wax on the five pairs of hearts is arranged to give a swollen and contractile appearance. The alimentary canal is conveniently represented by a cardboard cylinder enamelled white. (Several coats of "Luc" enamel will do this adequately.) If desired, a section in the middle of the model may be left with uncovered wire to indicate the omission of this region.

*A Simple Apparatus for illuminating Gelatine Models.*  
—A wooden box about 3 in. deep is used. The bottom is removed and replaced by a sheet of glass. A hole is

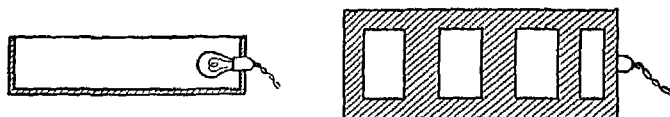


FIG. 6.

bored at one end to hold an electric light adapter and the interior of the box lined with white asbestos. The glass cover is covered with black paper, which is cut out to receive the cells containing the models, which then stand out very clearly.

## 10. MODEL OF A SNAKE'S SKULL

*Ian T. Hamilton*

It is not altogether an easy matter to represent the action of the movable quadrate in the skull of snakes by

the aid of diagrams alone, and although actual skulls may be available, they are so prepared that the quadrate is immovably attached to the skull and to the lower jaw.

Working models can easily be made in cardboard or three-ply wood. Except that cardboard is less durable, it is a better material for making the model.

The idea of the model is not entirely original, nor is

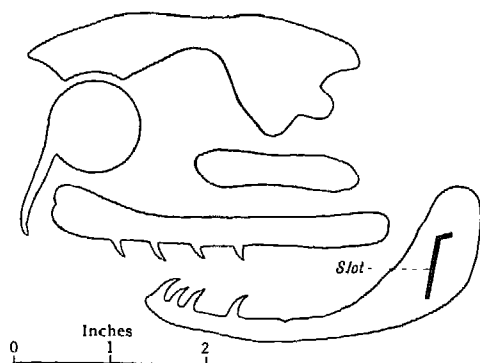
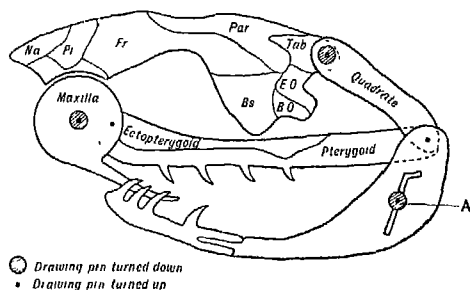


FIG 7.

any claim made that it is constructed accurately: it illustrates the principle of the mechanism whereby the gape is made unusually large, and at the same time, the poison fang is made to open outwards as the mouth opens.

The accompanying diagram is made to scale, and needs only to be cut out in stiff cardboard, assembled, and fixed to a base board. The chief difficulty in getting the model

to work is finding the position of the pin A. A suitable position for this pin is suggested on the diagram. By varying the position of the pin, a gape of varying size may be obtained. This pin is, of course, an artificiality, and does not exist in the animal: it is necessary for the working of the model, however, but can be covered over with a piece of paper.

It has been found impracticable to make the model more vivid by inserting muscle attachments with elastic bands. All that is required to open the mouth is a push downwards at the point of articulation of the quadrate with the lower jaw

## 11. PHOTOMICROGRAPHY WITH A BOX FILM CAMERA

*K. J. Savage*

On page 236 of *The Science Masters' Book*, Series I, part 1, is described a method of taking photomicrographs with a camera from which the lens has been removed. The method necessitates the use of plates and requires a focusing screen. The following apparatus, made from an old box film camera and a couple of old microscope eyepieces, may be of interest. Here the real image formed by the microscope objective is cast straight on to the photographic film. Several photographs are taken on one roll film; focusing for each photograph is done with a device separate from the camera. No dark-room is required, and the method requires no more knowledge of photographic technique than is necessary to take snapshots.

The camera actually used was a Zeiss Ikon Baby Box Tengor, which takes 16 small photographs on a V.P.K. film, but presumably any other cheap box camera could be adapted. Lens and shutter mechanism are removed and the tube of one of the old eyepieces is soldered on in front, as shown in the diagram. The hole marked A has

to be enlarged somewhat, and a simple shutter is placed in front; this consists of a piece of tin which can be pulled in front of the hole by a couple of pins projecting through the side of the camera (in the case of the Box Tengor two small holes were left in the right positions when the wire view-finder was removed) The front lens of the eyepiece is converted into a cap which screws into the tube when the instrument is not in use, thus eliminating any possibility of the entrance of light. By the time these alterations have been made, it will be necessary

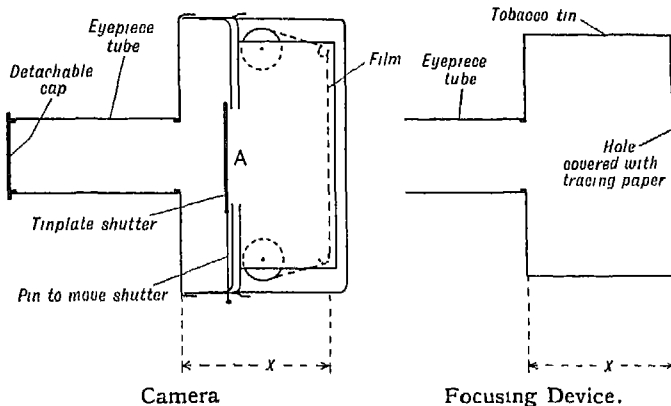


FIG 8.

to give the inside of the camera another coat of dull black paint.

The focusing device consists of a round  $\frac{1}{4}$ -lb. tobacco tin with a hole cut in the bottom. The second eyepiece tube is soldered round this hole, as in the case of the camera. The lid of the tin has another larger hole cut in it, and over this hole is stuck a piece of tracing paper to act as a focusing screen. The tin is then cut down in length so that the distance from its bottom end to the focusing screen exactly equals the distance from the front of the camera to the film at the back (distance marked  $x$  in the diagram). The inside of the tin should be given a coat of black paint and the lid soldered into position.

The microscope used should be fitted with a condenser and iris diaphragm. The method of using the apparatus is as follows :

(1) Slip the focusing device into the tube of the microscope in place of the usual eyepiece. Illuminate the slide strongly, using the condenser, and focus until a clear image of the slide is seen on the tracing-paper screen. See that the whole field is evenly illuminated (this is the most troublesome operation, and requires a certain amount of manipulation of the source of light and of the condenser).

(2) Close the iris diaphragm so that no light gets through to the slide (if the condenser is fitted with a ring for holding filters, etc., a dark stop can be placed here as an additional precaution), and replace the focusing device by the (loaded) camera. Open the shutter of the latter. Since the length of the tobacco tin was made exactly equal to the distance from the front of the camera to the film, the image will be in focus on the film.

(3) To make the exposure, open the iris diaphragm of the microscope for the desired interval of time. When the exposure has been made, close the shutter of the camera, replace the tobacco tin and focus up for the next slide.

The apparatus is most conveniently used in a horizontal position, but for temporary preparations, etc., it can be used vertically (the mirror will be removed for horizontal work). Using 18° Sch. films, exposures are of the order of 2 secs. with a 2 in. or  $\frac{3}{4}$ -in. objective, and somewhat longer with a  $\frac{1}{6}$ -in. (some difficulty may be found in getting even illumination with this objective). A filter in front of the condenser usually produces clearer results ; what colour to use naturally depends on the staining of the slide, but a red (Chance-Watson no. 2) and a green (no. 5) have been found very useful. Filters have the added advantage of making the exposure longer.

The whole apparatus is easy to make and cheap to use, since 16 exposures can be made on one film. The only operation in the making which requires much care

is the cutting down of the tobacco tin to the right length. Whether this has been done properly can easily be tested by slipping a strip of tracing paper into the camera in place of the film and comparing the image formed on this with that formed on the focusing screen. The negatives are small, but in these days, when miniature cameras are so popular, this cannot be regarded as a serious drawback, since enlargements are easily made or obtained.

## ANIMAL HISTOLOGY

### 12. HISTOLOGICAL TECHNIQUE

*T. L. Green*

The study of animal structure entails microscopic examination of sections or whole mounts. Both of these are prepared by fundamentally similar techniques in which the essential stages are as follows :

<i>Whole mounts.</i>	<i>Sections.</i>
1. Killing.	1. Killing.
2. Fixing	2. Fixing
3. Staining.	3. Dehydration.
4. Dehydration.	4. Embedding.
5. Clearing.	5. Sectioning
6. Mounting.	6. Staining.
	7. Final dehydration.
	8. Clearing.
	9. Mounting.

As far as schools are concerned, the students are generally concerned with whole mounts only. They may perhaps make some hand sections, but it is unlikely that such work would appear in an examination. For this reason the following notes will not deal with microscopic technique, for this see D. M. Reid on "Histological Methods" in the *Science Masters' Book*, Series I, part 2, p. 186.

*Killing.*—The method adopted obviously depends upon the animal concerned. Small organisms are dropped into the fixative at once; larger ones are anæsthetized with chloroform, coal gas, etc. Even small organisms

may have to be anaesthetized before fixation in order to prevent excessive twisting and deformation. This is often necessary in the case of insect larvæ, especially aquatic ones. For the latter a few crystals of menthol should be dropped upon the water surface. For hard-bodied beetles and some animals such as the earthworm, momentary immersion in boiling water may be tried with success.

*Fixation.*—After rapid killing comes fixation, by which means the general structure of the animal, both microscopic and macroscopic, is preserved as nearly as possible as it was in life. In addition, fixation hardens the object, so that it can be handled without disintegration occurring. Finally, by the action of fixatives, we not only form insoluble compounds with the cell constituents, but also bring about an optical differentiation of the constituents so that they are more easily seen. Some fixatives also act as mordants preliminary to subsequent staining.

The following rules should be rigidly observed.

(a) Be sure the tissue is as fresh as possible so that no post-mortem changes have taken place.

(b) Be sure the fixative is in perfect working order, which generally means freshly made, because some solutions do not keep.

(c) Use a large volume of fixative relative to the tissue, which should always be as small as possible to facilitate penetration.

(d) Fix for the optimum period of time which is a function of several factors, such as temperature, size of tissue, rate of penetration, and which must generally be decided by experiment.

(e) Wash out the fixative with appropriate means when fixation is complete; this avoids "over-fixation," the precipitation of crystals, etc., in the tissue.

The choice of a fixative depends upon the results desired, but in general school work any of the following may be used:

*Bouin's Solution*.—For all general histology.

Saturated aqueous solution of Picric acid	75 parts
Formol (40 per cent. formalin) . . .	25 „
Acetic acid . . . . .	5 „

Fix for periods between six and eighteen hours (e.g. small piece of earthworm six hours, small frog tadpole twelve hours, chick embryos twelve hours, largest tadpoles eighteen hours).

Wash out the picric colour in several changes of 70 per cent. alcohol. It need not be all removed

*Formalin 10 per cent.*—For pieces of nerve tissues, the brain, etc. Fix for twelve hours, below room temperature if possible, and finally preserve in 2½ per cent formalin.

*Flemming's Fluid*.—For cytological work in which it is desired to demonstrate fat globules, mitochondria, phases in nuclear division, etc. A difficult and expensive fixative

1 per cent. chromic acid	. 15 parts.
2 per cent osmic acid	4 „

Fix very small pieces of tissue for twelve hours and wash thoroughly in running water for twelve hours.

After fixation, material should be stored in 70 per cent. alcohol in stoppered bottles or corked tubes; the ends of the latter should be dipped in wax to prevent evaporation.

*Staining*.—Animal tissues are stained in various ways, so that different cellular elements may be distinguished. Stains may be “specific” and act only on one cell constituent, or “general” and stain the whole cell. They are also classed as “nuclear” or “plasma” stains for other obvious reasons. A stain may be used alone or with a second counter-stain.

In practice, there are two modes of staining—simple, direct, or progressive staining, and indirect or regressive staining.

In the case of the former, the object is merely placed in the stain until it reaches the desired colour and then



at once mounted. In regressive staining, the object is considerably overstained and subsequently "differentiated" to the desired degree. As a result of this method, and because the cell elements have different affinities for the stain, it will be found that the various cell elements are more clearly defined than is the case in simple progressive staining.

Among the multiplicity of stains which are available, it will be found that the following brief selection will do everything that is necessary for a school course.

*Borax Carmine.*

4 per cent. aqueous borax	.	.	100 c.c.
Carmine	.	.	1 gm
70 per cent. alcohol	.	.	100 c.c.

The last item is not added until the carmine has been completely dissolved by boiling in the borax solution. Filter when cold.

*DeLafield's Hæmatoxylin.*

(a) Dissolve 4 gm. of hæmatoxylin crystals in 25 c.c. of 90 per cent. alcohol

(b) 400 c.c. of saturated aqueous ammonia alum.

Mix (a) and (b) and stand exposed to air for 3 days; then add 100 c.c. of glycerine and 100 c.c. methylic alcohol

Stand for a week, then filter. Fit to use after standing a month or so. Keeps for years. Dilute with water (about 1 to 5 water) for use.

*Ehrlich's Hæmatoxylin.*

Hæmatoxylin	.	.	.	2 gm
Glacial acetic acid	.	.	.	10 c.c.
Glycerine	.	.	.	100 "
Absolute alcohol	.	.	.	100 "
Water	.	.	.	100 "
Ammonia alum	.	.	.	to saturation.

Keep in light and open to the air until the stain ripens to a dark red colour.

All the previous three stains are used for the regressive method and differentiated with acid 70 per cent. alcohol,

about 0.5 per cent. HCl in 70 per cent. alcohol. The hæmatoxylin stains must be neutralized or "blued" with ammonia after differentiation, and the correct sequence is :

Stain.

Wash in 70 per cent alcohol.

Differentiate in acid 70 per cent alcohol (about one minute).

Wash in 70 per cent. alcohol (one minute).

Wash in 90 per cent. alcohol (one minute).

"Blue" over ammonia bottle or in alkaline 90 per cent. alcohol

Then dehydrate and mount.

If this sequence is shortened, i.e. if the material is taken at once from acid alcohol to alkaline alcohol, there will be a white flocculent precipitate.

In the case of the two hæmatoxylin, it may be considered that a counter-stain would be an improvement. For this purpose the following may be used with success :

Alcoholic Eosin	. . . . .	1 per cent in absolute alcohol.
Orange G or Light Green	. . . . .	0.5 per cent in absolute alcohol.

*Mallory's Triple Stain.*—This stain necessitates the use of phosphomolybdic acid as a mordant, but by using the following formula, it is not necessary to use the mordant and stain separately.

A.	0.2 per cent Acid Fuchsin.	
B.	Aniline blue	0.5 gr
	Orange G	2.0 "
	1 per cent Phosphomolybdic acid	100 c.c.

Stain for one minute in A, wash in water and then stain in B for about ten to thirty minutes. Differentiate in weak alcohol, rapidly dehydrate and mount in balsam.

Brilmyer's modification of Mallory is first to stain the material in Delafield's hæmatoxylin for five minutes and then follow the technique as for Mallory. This method shows up the nuclei particularly well.

*Heidenhain's Iron Hæmatoxylin.*

- |    |   |          |
|----|---|----------|
| A. | Iron ammonium alum  | 5 gm.    |
|    | Distilled water   | 100 c.c. |
|    | (Use only crystals of pale amethyst colour.)  |          |
| B. | Hæmatoxylin   | 0.5 gm.  |
|    | Absolute alcohol  | 10 c.c.  |
|    | Distilled water   | 90 "     |
|    | (Dissolve Hæmatoxylin in alcohol, add water, and allow several weeks for ripening.) |          |
| C. | Iron ammonium alum  | 2 gm.    |
|    | Distilled water   | 100 c.c. |

Immerse sections in A for half to one hour; drain off the excess and stain in B for one to two hours. Differentiate with C, *under the microscope*.

*Heidenham's Hæmatoxylin (Anderson's Method).*

This may be used instead of the above when it is necessary to carry through the process in a short time.

- |    |  |          |
|----|--|----------|
| A. | Saturated solution of potash or ammonium alum in boiling water. Cool and filter. |          |
| B. | Shake up 2 gm of bleaching powder in 100 c.c. of water and filter.               |          |
| C. | Hæmatoxylin  | 0.25 gm. |
|    | Absolute alcohol   | 5 c.c.   |

(Add C to 20 c.c. of B and add the whole to 70 c.c. of A, together with 5 c.c. of Glacial Acetic Acid.)

Stain for one to five minutes.

*Methylene Blue.*

- |    |                 |          |
|----|-----------------|----------|
| A. | Methylene blue  | 1 gm.    |
|    | Distilled water | 100 c.c. |
| B. | Silver nitrate  | 0.5 gm.  |
|    | Distilled water | 100 c.c. |

(Heat B to dissolve, and slowly add 3 per cent. potassium hydroxide until no more precipitation occurs. Collect and wash the precipitate of silver oxide and add it to A. Boil and allow to cool.

For use, dilute with 4 parts of distilled water.)

Stain the material on a slide for several minutes, heating gently over a Bunsen flame. Allow to cool and rapidly differentiate with 95 per cent alcohol.

*Weigert's Stain.*—The successful preparation of this stain is not easy ; it should be bought and made up for use according to the maker's instructions

Stain the material for about thirty minutes. Wash in 70 per cent and 90 per cent alcohol and mount, or counter-stain, as required.

### *Safranin*

Safranin	.	.	.	.	.	1 gm.
96 per cent. alcohol	.	.	.	.	.	52 c c
Aniline water	.	.	.	.	.	48 c.c

To prepare aniline water, shake up 10 c.c. of aniline in one litre of hot water. Filter when cold.

As an alternative, use a 1 per cent. aqueous solution of safranin. Stain for a few minutes, wash in alcohol, dehydrate, clear and mount.

Very useful as a counter-stain.

### *Orange G.*

Any of the following formulæ may be used as convenient :

- (a) Saturated soln. of Orange G in absolute alcohol.
  - (b) Saturated soln. of Orange G in clove oil
  - (c) Orange G . . . . . 0.5 gm.
  - Distilled water . . . . . 100 c.c
- (Use as a counter-stain.) (Table I.)

### *Eosin.*

- Either* (a) Eosin (alcohol soluble) . . . . . 1 gm.
  - 70 per cent alcohol . . . . . 100 c c.
  - or* (b) Eosin (water soluble) . . . . . 1 gm.
  - Distilled water . . . . . 100 c c
- (Use as a counter-stain ) (Table I )

*Dehydration.*—It is generally essential to remove all water from the tissues by complete dehydration. For this purpose, solutions of alcohol up to absolute strength are employed, the tissue or slide being moved up from one to the other. The chief faults evinced by students up for examination are :

1. Failure to allow enough time in each concentration.

2. Failure to give two or more changes of absolute alcohol.
3. Failure to minimize exposure to air as much as possible in making the change.
4. Failure to keep the 100 per cent. alcohol bottles stoppered or leaving material in absolute in an uncovered watch-glass.
5. The habit of using forceps or brushes charged with low-grade alcohol (or even water!) to handle material in the final stages.

*Clearing.*—Having removed all water from the object, it must be “cleared,” a process which both renders it transparent and also introduces a fluid miscible with Canada balsam, etc. The chief reagents are: Cedar-wood oil, Clove oil, Xylol. These are all good, but note that in xylol delicate objects (such as medusæ and nephridia) shrink and become crumpled, and that clove oil renders tissues brittle.

Xylol will clear only a completely dehydrated object.

Cedar oil will clear from 96 per cent. alcohol, but is not always to be relied on for this capacity.

Clove oil will clear from 94 or 95 per cent. alcohol.

By using Terpeneol, tissues can be cleared from 90 per cent. alcohol, which then obviates the use of expensive absolute alcohol.

Whenever possible, use the diffusion gradient method.

*Mounting.*—The final stage in the preparation of a specimen is the mounting. The mountants used include:

- (i) *Glycerine Jelly* may be used and does not require dehydration. This may be made as follows: Take 1 part of best French gelatin and soak for three hours in water. Pour away the superfluous water and heat till melted; to this add the glycerine. Add a drop or two of carbolic acid.

When using this for mounting, the stock mass is warmed till melted, the object is placed in a drop of water in a watch-glass and some glycerine added. It

is then placed in a drop of the warmed jelly till impregnated, and finally placed upon a slide and a cover-glass added.

(ii) *Glycerine* itself can be used in a celled slide. The object is in water, to which more and more glycerine is added. It is then transferred to pure glycerine on the slide. Such a slide should be "ringed" with varnish, gold size or asphaltum.

(iii) *Canada Balsam*.—This is best dissolved in xylol, though a benzol solution will set most quickly. Objects must be thoroughly dehydrated. The freshly made slide must be kept in a warm oven until hard. Balsam supplies in the school laboratory should include "thin," "medium" and "thick" solutions for various purposes.

The slide and cover-slip should be washed clean in hot soapy water, rinsed in acid 70 per cent alcohol and dried carefully with a soft clean rag. On a sheet of white paper make a  $3 \times 1$  in. rectangle and draw the diagonals. Place the slide on this for centering purposes. Place a drop of balsam on the slide, add the object and some more balsam. Use the minimum. Allow it to spread slowly. Stand the cover-glass in position over the object, resting on one edge and supported by a needle below the other side. Lower gently into position. Re-centre if necessary. Dry hard. Scrape away any superfluous balsam and wipe down with absolute alcohol. Label and record name of object, fixative, stain (and counter-stain if used), date made and name of maker. Run a ring of copal varnish or white cellulose paint round the edge of the cover-glass.

*Whole Mounts*—Details are given below from actual laboratory preparations of "whole mounts" such as are often set in examinations. Of course the times in solution are obviously subject to variation, and the following can only be a rough guide.

## TYPE SPECIMEN METHODS

*A Smear from Vesicula Seminalis of Earthworm to show Monocystis.*

- (i) Clean six cover-glasses and six slides.
- (ii) Have ready at least 12 watch-glasses.
- (iii) Dissect out vesicula and smear the cut surface very lightly on the cover-glasses.
- (iv) Allow the smear nearly to dry, exposed to the air.
- (v) Fix in 70 per cent alcohol for five minutes.  
Handle with forceps and drop each glass smear upwards into a watch-glassful of alcohol.  
Cover with a second watch-glass.
- (vi) Stain in Ehrlich's hæmatoxylin for fifteen minutes.
- (vii) Wash in 70 per cent. alcohol.
- (viii) Differentiate in acid 70 per cent.—five to twelve minutes—till of a pale reddish colour.
- (ix) Wash in 70 per cent., wash in 90 per cent., then "blue" in ammonia vapour.
- (x) Dehydrate, 90 per cent., five minutes; two changes absolute, five minutes each.
- (xi) Clear in xylol.
- (xii) Mount on spot of balsam on slide. Be careful to reverse the cover-glass and get the smear downwards

*Ovary of the Earthworm.*

- (i) Wash and dry slides and slips, also a pipette.
- (ii) Have glycerine and watch-glasses ready.
- (iii) Dissect out ovary and examine with microscope in water; remove all traces of septum, muscle, etc.
- (iv) Mix a drop of water and a drop of glycerine and transfer ovary to this in a pipette; five minutes.
- (v) Add glycerine drop by drop; fifteen minutes.
- (vi) Place drop of glycerine on slide. Into this place the ovary and cover with glass slip.

If a ringed slide or a slide with a cell ground out is not available, it is wise for the pupil to cut out a paper square the size of the cover-glass. Fold this and cut out the centre. Open out flat and soak in water and then in glycerine till wanted. Place on the slide with ovary in the middle. The thickness of the paper will prevent crushing of the ovary.

It is sometimes necessary to stain an object and then mount it in glycerine. In this case stains in aqueous solution are used in the progressive fashion, or an alcoholic stain may be used regressively and the object subsequently brought down to water before mounting.

By varying the above methods, any tissue should be easily dealt with. The great thing is to have all the necessary apparatus ready first. Then dissect out from the freshly killed animal the required tissue and fix it; for all pupils' work alcohol will do. If it can be stuck to a cover-glass for a support, so much the better.

#### *Cartilage.*

Prepare a slide of cartilage, using a single stain.

1. Make a thin slice of cartilage
2. Fix in 70 per cent. alcohol, fifteen minutes
3. Stain in Weigert for thirty minutes.
4. Wash in 70 per cent. and 90 per cent. alcohol, five minutes.
5. Complete dehydration in two changes 100 per cent. alcohol, ten minutes.
6. Clear in clove oil, five minutes.
7. Mount in balsam.

If a counter-stain is required (such as Eosin or Orange G), it must be made up in absolute alcohol or clove oil, and will thus act during dehydration or clearing. If staining is complete before dehydration or clearing, remove to a second bath of the appropriate fluid minus the stain.



STANDARD HISTOLOGICAL METHODS FOR COMMON TISSUES

Tissue.	Locality	Fixative	Stains		Appearance	Mountant
			Major.	Minor.		
Cartilage	Nasal capsule or joint of frog Skull of dogfish	10 per cent Formalin 3 per cent acetic acid Boun's fluid	Delafeld's Hematoxylin and Eosin Weigert's stain		Ground substance blue to purple Shows up elastic fibres	Temporary— Glycerine Permanent— Gurr's Neutral Mountant Canada balsam
Muscle—Striped Unstriped	Leg muscle, etc { Coat of Intestine { Bladder	5 per cent Formalin in 1 per cent NaCl Saturated aqueous Mer- curic Chloride Boun's fluid	Hendehain's Iron Hæ- matoxylin Delafeld's Hematoxylin and Eosin		Shows up all detail well Sarcomplasm pinkish Sarcomerema bluish Nuclei blue	Canada balsam
Chabated epithelium	Lung of frog's mouth	5 per cent Formalin 70 per cent Alcohol	Delafeld's Hematoxylin and Eosin Borax carmuine		Nuclei blue Cytoplasm red Nuclei red Cytoplasm pink	Glycerine Canada balsam
Nervous Tissue	T S spinal cord, pieces of nerve, etc	10 per cent Formalin Sat aq HgCl <sub>2</sub> 90 per cent alcohol Boun's fluid	Methylene blue Hendehain's Iron Hæ- matoxylin Borax carmuine		General stain Shows Nissl gran- ules General stain Nuclei black Carmuine is good for non-nervous elements	Glycerine Canada balsam Gurr's Neutral (Preferably one of the last two)
Tendon	Tendinous attach- ments of frog's muscle	5 per cent. Formalin 70 per cent. alcohol	Weigert and Safranin Mallory's Triple Stain		Fibres blue to black Nuclei red Ground substance yellow Elast fibres reddish Conn. tiss. blue Conn. tiss. fibres blue Nuclei red	"
Pigment Cells	Frog skin	Absolute alcohol 5 per cent Formalin	Mount some skin un- stained in <i>glycerine jelly</i> Iron-hematoxylin and Orange G Delafeld's Hematoxylin and Eosin Mallory's Triple Stain		Cytoplasm orange Cytoplasm red Connect tissue and derivatives blue Mucin and glands blue Blood corpuscles yellow. Pig- ment (melanin) show black	Glycerine Canada balsam

## PREPARATION OF BLOOD FILMS

To make a good slide of blood requires :

- (i) An absolutely clean glass surface ;
- (ii) A thin even film of blood—not a “ smear ” ;
- (iii) Suitable preliminary treatment before staining ;
- (iv) Considerable practice in each method.

The most usual forms of technique for this work are given in Table II. Further complete formulæ must be referred to in textbooks. The following are the general precautions :

Slides must be cleaned in hot soap and water, washed in acid-alcohol and dried, after dipping in 90 per cent alcohol, with a soft clean cloth

The blood film is made by taking a small blood drop on the edge of a cover-glass or slide ; this is held at 45° to the surface of the other glass and then drawn slowly along. It should leave a translucent film which is one cell thick

Blood films are generally air dried before fixation. The drying must be rapid (hence waving in the air, etc.), otherwise shrinkage and crenation of the corpuscle will result.

## TYPE SPECIMEN METHOD

To prepare a blood film of frog's blood, double-stained in methylene blue and eosin.

1. Collect blood with fine hard glass pipette—from any artery or vein or even from the heart.
2. Make a thin film on a slide or cover-glass as already described.
3. Air dry by waving about rapidly. Correct estimation of the degree of drying is a matter of experience, and also seems to depend upon the technique to be used later. It is well to make two or three films and dry to different extents and then select the most suitable result.
4. Fix in absolute alcohol for ten minutes.

METHODS FOR BLOOD FILMS

Technique.		Results.			
Fixative	Staining Method.	Erythrocytes	Leucocytes	Platelets	Nuclei.
Vapour of 40 per cent. Formalin	Delafeld's Hæmatoxylin and Eosin	Red	Blue	Pink	Blue
(i) 10 parts Formol (ii) 90 parts 100 per cent. Alcohol (iii) Absolute Alcohol	Methylene blue and Eosin	Red-pink	Blue-purple	Pinkish	Bluish
Do not fix at all. . . .	Leishman's Stain (Methylene blue, Methylene Azur and Eosin)	Orange-pink	Blue	Purple	Blue
Absolute alcohol . . . .	Giemsa (As Leishman, but acts more slowly)	Bluish	Deep blue	Purple	Purple
Absolute alcohol . . . .	Laveran's Eosin and Methylene blue	Pink	Blue-purple	Pink-purple	Bluish

5. Stain for three minutes in saturated eosin in 70 per cent alcohol.
6. Wash in 70 per cent. alcohol
7. Stain in 1 per cent methylene blue in 70 per cent. alcohol for one minute.
8. (a) Dehydrate, clear and mount in balsam.  
      (b) Wash in water and mount in glycerine jelly.  
      (c) Wash in alcohol and water, blot and store as a dry film.

### 13. METHOD FOR STAINING SMEARS OF SEMINAL VESICLES OF EARTHWORM

*Department of Zoology, Glasgow University*

Excellent preparations may be obtained under ordinary class conditions by the following method, for which the writer is indebted to the Department of Zoology of Glasgow University :

The contents of the seminal vesicles of a *freshly killed* worm are examined for the presence of the trophozoites of *Monocystis*. If these are found, a *very thin* smear is made on a clean cover-slip ; this is immediately dropped face downwards into a watch-glass, containing a saturated solution of corrosive sublimate to which has been added acetic acid (1 c.c. of glacial acetic to 100 c.c. of the solution ) It is important that the smears should be thin, or much of the material will wash off In the subsequent proceedings, the film must on no account be allowed to dry. Schedule :

1. Pour off the fixative into the bottle.
2. Wash successively in (1) water, (2) 30 per cent. alcohol, (3) 50 per cent. alcohol, (4) 70 per cent. alcohol, to which has been added a few drops of iodine solution (this removes the last traces of the fixative), (5) plain 70 per cent. alcohol.
- 3 Stain in Borax Carmine for ten minutes.
4. Give a quick rinse in 70 per cent. alcohol and differ-

entiate in acid 70 per cent. alcohol (1 or 2 drops of concentrated hydrochloric or nitric acid to 100 c.c. of 70 per cent. alcohol). Care must be taken to avoid overdoing this stage. The film should still be a very bright pink when removed.

5. Wash thoroughly but quickly in 70 per cent. alcohol.

6. Dehydrate for five minutes in absolute alcohol.

7. Clear in clove oil and mount in Canada balsam.

In the finished preparation, the larger trophozoites of *Monocystis* stand out well with their nuclei clearly defined. The smaller ones inside the sperm morulae need searching for under the high power. The method of fixation employed gives better results than the more usual one of fixing a partially dried smear with absolute alcohol, since the degree of shrinkage is much less. Moreover, staining with hæmatoxylin rarely gives satisfactory results in the short time available in the ordinary class periods.

The class should be warned of the very poisonous nature of the fixative and of the necessity for not allowing it to come into contact with the fingers or with metal forceps. If any difficulty is experienced in handling the cover-slip, it may be gripped in a small split in the side of a wooden splint at the commencement of the operations.

### ANIMAL MORPHOLOGY

#### 14. MACROSCOPIC METHOD OF DEMONSTRATING DORSAL PORES OF EARTHWORM<sup>1</sup>

*S. Oram*

The usual method of demonstrating the dorsal pores of *Lumbricus* to a class, by wiping the worm and then squeezing it to cause exudation of coelomic fluid, is admittedly unsatisfactory. The following method is simple and effective.

<sup>1</sup> From the Zoology Department, King's College, University of London.

Freshly killed worms should be used. The animal is killed by immersion in methylated spirit. This avoids peeling of the cuticle, such as tends to follow the use of chloroform. A piece about 2 cm. long is cut off. Either anterior or posterior parts work equally well, although, of course, there are no dorsal pores in the first seven or so annular grooves of the anterior end. One worm will provide material for two demonstrations ; and since only

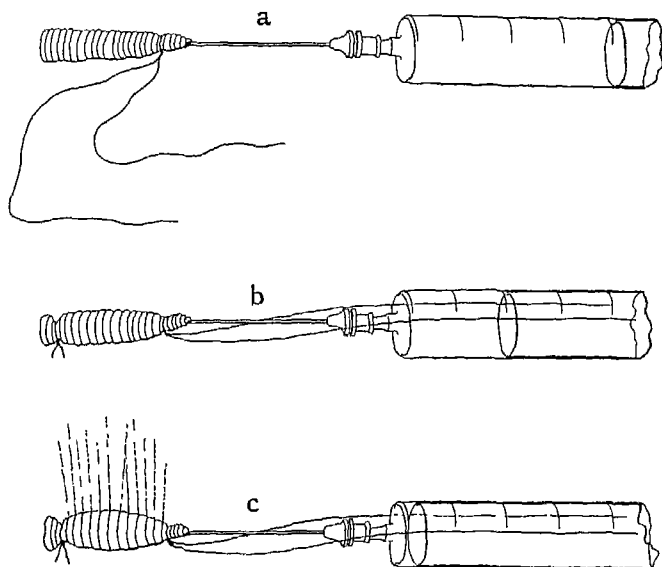


FIG. 9

the anterior half is usually required for dissection by a class, the unopened hind end can readily be utilized if material is scanty.

A 20-c.c hypodermic syringe, previously filled with some coloured solution such as eosin, is carefully introduced into the gut of the worm, either through the mouth or the anus, and the needle is pushed about 0.5 cm. along the pharynx or intestine. Care is taken not to puncture the gut wall. Then a ligature is tied

behind the needle point and the ends of the ligature are left about a couple of inches in length (*a*). Next, part of the solution is carefully injected: this first injection washes the gut free from most of its contents and generally requires about 10 c.c. of fluid. Then, without removing the needle, tie a second ligature distal to the first (*b*). Now the two long ends of the first ligature are held firmly against the barrel of the syringe, and the remaining volume of eosin is *forcibly* injected (*c*).

At first the piece of worm swells and tends to slip from the needle, but the slipping is prevented by gripping the ends of the first-tied ligature, as explained. In a short time, the presence of dorsal pores is evidenced by very fine jets of coloured fluid, which spray out from them and are plainly discernible against a background such as a white dissection-dish.

Examination of the worm after the experiment shows that the forcibly injected fluid has ruptured the gut, mainly along its ventral side, and has found its way into the coelom, the successive portions of which between the septa become severally filled and set in communication. Thence the fluid is forced out through the dorsal pores.

## ANIMAL PHYSIOLOGY

### 15. PHYSIOLOGY OF THE HEART

*G. N. Ridley*

The method is briefly that of transferring, with magnification, the pulsations of the heart by a light lever to a recording surface. The lever (cardiograph) used in this case is a thin strip of wood, about 40 in. long (longer than the usual cardiograph but having the advantage of movements visible to a class). Strip aluminium is also satisfactory. A pointer of paper fixed at one end provides a "pen." For the fulcrum, a glass T piece is used, the strip being fastened into the long tube, the tube at

right angles to this being passed over a thin glass rod held in a clamp. A weight of about 20 grams hangs at a suitable position from the short arm of the lever to take the weight of the long arm off the heart, which is attached by thread from a thin hooked pin passed through the apex of the ventricle.

The recording drum is an ordinary clockwork klinostat. The fixed rate of rotation (one revolution per fifteen minutes) naturally limits the type of experiment performed, but enables satisfactory cardiograms, for comparative purposes, to be made. There is no sub-

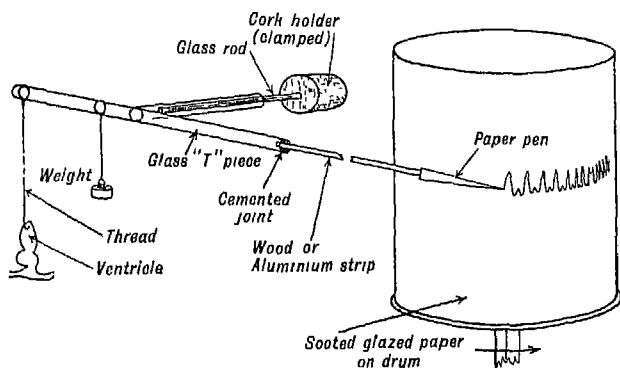


FIG 10.

stitute for the special glazed recording paper which is sold for this purpose; it should be fastened around the celluloid cover of the klinostat and smoked evenly.

*Preparation.*—The frog is killed with chloroform, washed and pinned out on a cork platform held at the required height by a retort stand. The animal is opened ventrally, the heart exposed and freed from the pericardium. The heart is then attached to the short arm of the lever by the hook and thread, the tension on the organ being just sufficient to stretch it slightly. The rhythmic movements of the paper pen are then recorded by bringing the smoked paper carefully up to it.

*Tracings*—The first small rise of the lever is due to



the auricular contraction or systole, the second larger rise to the contraction of the ventricle. The series of pulsations constitutes the *cardiac cycle* which, fully expressed, in the frog, is in the order: sinus venosus, auricles, ventricle, truncus arteriosus.

*Effect of Heat*—A tracing of the normal beat, the heart being bathed with Ringer's solution at room temperature, is recorded first and at intervals Ringer's solution at 25° C. applied to the heart. The marked response by augmented ventricular contraction and general muscular force is clearly shown. Ringer's solution at 40° C produces a difference in response, the systole being much reduced and the rate of pulsation enormously increased.

*Effect of Potassium*—A small excess of potassium chloride is added to the Ringer's solution. The contractions are diminished and the heart stops in diastole.

*Effect of Sugar*.—Ringer's solution containing 1 gram per litre of glucose is substituted for the ordinary fluid. No marked effect is recorded. (This concentration of glucose is used in perfusion fluid for the mammalian heart. Increasing strengths of glucose might be investigated.)

*Effect of Alcohol*.—Weak solutions of alcohol in Ringer's solution cause a definite stimulation of the heart-beat.

*Effect of Adrenalin*—Adrenalin is extracted from the suprarenals of a rabbit by grinding them up with about 15 c.c. Ringer's solution, boiling and filtering the fluid. This is diluted to three or four times its volume. On applying this extract to the heart, a characteristic increase in ventricular systole is obtained which is removed by rinsing with plain Ringer's solution. When adrenalin is passed into the heart with the perfusion fluid (in the living animal, with the *blood*), it causes both an increased force and rate, but there is no indication of the latter change where the hormone is in contact only with the surface of the heart.

To preserve the tracings the paper is cut across, passed through shellac varnish and allowed to dry.<sup>1</sup>

## 16 INVESTIGATION OF THE EFFECT OF THYROID ON THE DEVELOPMENT OF TADPOLES

A. K. C. Ottaway

The main stock of tadpoles was obtained from a local, well-known pond, where the eggs were laid in the third week of March. All the tadpoles used were from the same batch of eggs in order to ensure equal genetic conditions. Until they were about six weeks old the tadpoles were kept in a large aquarium, and fed mostly on vegetable food, with an occasional meal of meat. As usual, they fed voraciously, and seemed very fond of filamentous weed, like *Spirogyra*.

We now had a stock of tadpoles in healthy condition and all as nearly as possible at the same stage of development. How then to test the suggestion that we can change the rate of development of these tadpoles at will, by the simple means of changing their diet? It will be easily appreciated that the first essential is to keep a certain section of the stock under perfectly normal standard conditions, so as to compare them with others treated in the same manner, but with the addition of the special factor under investigation. Such a section is called a Control Group. A checking experiment of this nature is essential to any true interpretation of the results, and it requires as much care and treatment as any other. Although its use may seem obvious, it is necessary to emphasize the importance of the Control Group, since it is the chief feature of this particular experimental technique, and is an example of the way in which Biology is now employing recognized methods of Physics and Chemistry.

<sup>1</sup> Reproductions of the tracings actually obtained will be found in the original article, *School Science Review*, xiv [53], 68-70.

At the age of six weeks, the main stock of tadpoles was divided into two groups .

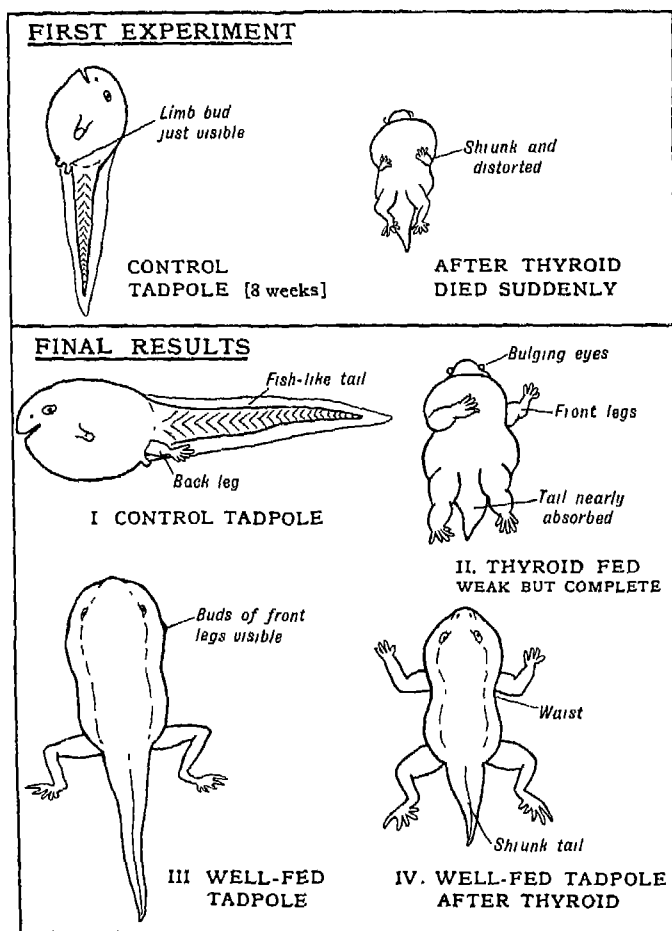


FIG. 11.

(1) Control Group, to be fed normally as vegetarians, with a small amount of meat food occasionally.

(2) Very Well-fed Group, given plenty of meat food

and vegetable matter, and less crowded feeding conditions.

Four weeks after this separation, that is, at the age of about ten weeks, the Control Group had developed back legs, while the Well-fed Group had their back legs well on, showed the front limb buds under the skin, and were much fatter than the Control.

Four Sub-Groups were now started from the two main stocks and labelled as follows :

- (1) Control Group.
- (2) Well-fed Group.
- (3) Thyroid A (Control Group plus thyroid).
- (4) Thyroid B (Well-fed plus thyroid).

These groups were isolated in round glass aquaria, about 10 in tall and 6 in in diameter. The aquaria were kept all together near a well-lit window inside the laboratory. Light and temperature conditions were thus kept pretty equal, and any change in the water was made to all at the same time. The water used was taken from the same pond in which the eggs were laid. Constant observation and attention had to be kept up throughout the period of the experiment. Dead bodies were immediately removed and care taken to prevent the water from being fouled in any way. Faeces were removed with a wide pipette to avoid changing the water too often.

Thyroid was administered daily by means of crushed tablets of sheep thyroid extract, each tablet being equivalent to 15 grains of the fresh gland. The daily dose was one-fiftieth of a tablet (i.e. one gram) per half-dozen tadpoles. The powdered extract was merely added to the water in the jar. It should be noted that the thyroid groups were also fed on their usual diet, the thyroid being added as an extra.

The results are shown in the diagram. This experiment being concerned with *rate of development*, dates and times should also be carefully noted. These will vary with local conditions. In the particular experiment

described, the Thyroid A Group metamorphosed in three days, but were small and weak. The Thyroid B (taken from the Well-fed stock) metamorphosed in under two days and became healthy little frogs. On killing a specimen from each group, it was possible to have an interesting collection of four tadpoles, all of the same age and from the same eggs, but in four different stages of development. Metamorphosis in the Control Group and the Well-fed Group did not occur until seventeen days and ten days later respectively.

## 17. CONDITIONS NECESSARY FOR EARLY METAMORPHOSIS OF TADPOLES

### *A. K. C. Ottaway*

From the Control Group described in the last experiment, tadpoles were taken at the age of eight weeks or under and given thyroid meals. The back limb buds were just visible in the Control. The Thyroid Group began to be very frisky, the back legs grew rapidly, and the front legs began to push through. The body began shrinking and changed shape. The eyes bulged and the branchial region became swollen and enlarged. Suddenly all of them died. The attempt at premature metamorphosis was too sudden; the increase of metabolism had wasted the tadpoles away. They were too young to stand up to such a rapid change of rate of development. The diagram shows the shrunk and distorted shape compared with the Control.

A similar result is obtained by giving an overdose of thyroid to tadpoles at a much later stage. A series of experiments can be planned to show that there is a relation between concentration of thyroid and rate of metamorphosis. The conditions necessary for successful metamorphosis before the usual time are that (1) thyroid should not be given at too early an age, and (2) that it should be given only in small quantities.

## ANIMAL TYPES

## 18. NOTES ON THE TREATMENT OF ANIMAL TYPES

*T L Green*

*Amœba*.—Kill by steam or dry heat or osmic acid vapour. Stain with Delafield's hæmatoxylin.

The preparation of slides is not easy and the methods generally adopted are complex. They are detailed in the standard textbooks of microscopical technique.

*Paramecium*.—As for *Amœba*. Borax carmine a good general stain. Iron hæmatoxylin and light green excellent for nuclear detail in conjugation

*Volvox* and *Stentor*.—Use a celled or cavity slide. Addition of glycerine will show cilia. Methyl green or methylene blue used *intra vitam* brings up cell limits clearly. Preparations may be unstained or stained in methyl green, etc. Mount in formol 5 per cent solution, or this with an equal volume of glycerine added.

*Vorticella*.—It is exceedingly difficult to avoid complete contraction. Menthol narcosis. Sudden osmic acid or heat fixation. Stain with Methyl, or Light Green, or Iron Hæmatoxylin and Orange G.

*Monocystis*.—Tease out seminal vesicles and receptaculum seminis of earthworm in 0.7 per cent. salt solution. Examine material at once in watch-glass—and treat worms one at a time! Trophozoites show slight "euglenoid" movements. Cysts should be crushed to show pseudonavicellæ.

Make thin smears on cover-slip. Fix in 90 per cent. alcohol after *nearly* drying in air. Stain in Ehrlich's hæmatoxylin. Differentiate thoroughly. Carry out all the operations in watch-glasses.

*Note*.—*Monocystis magna* may be found occasionally and is so large that it may be seen with the naked eye. It is difficult to stain and clear, but by using slow and careful methods success should result.

*Hydra*.—The cells comprising the tissues should be examined by maceration. Kill with 1 per cent. acetic acid with a trace of osmic acid. Leave in this solution for five minutes. Place in a watch-glass of water and disintegrate with a paint-brush.

To obtain mounts in expanded condition narcotize slowly with menthol. Sudden application of hot fixative (60° C.) may be tried—use Bouin or 10 per cent. Formalin.

Whole animals should be mounted in formol, glycerine or balsam. Sections should be stained in hæmatoxylin.

The nematocyst batteries may be shown in whole mounts by killing the specimen in hot saturated mercuric chloride in 70 per cent. alcohol, washing well in alcohol, staining in very strong methylene blue, rapidly dehydrating and clearing in cedar oil. The nematocysts are blue and the rest of the animal unstained.

*Obelia*.—If living material is available, examine both forms against a dark background.

(1) Hydroid. Narcotize living material with menthol. Fix in mercuric chloride, formol or alcohol. Stain in Borax carmine or Ehrlich's hæmatoxylin. (2) Medusa. Fix in mercuric chloride, formalin or alcohol. Prolonged overstaining and differentiation is necessary to show the otocysts. The otoliths are destroyed by the acid used for differentiation. Borax carmine is the best stain to use.

The chief troubles in dealing with the medusa are folding or crushing.

These may be avoided by rigidly observing three conditions.

(a) For picking up the medusa use a wide-mouthed pipette held vertically.

(b) Clear in cedar oil by the gradient method. Place the dehydrated medusa in a tube of absolute alcohol, tilt this to one side and run in cedar oil down the glass slope drop by drop. Cork the tube and allow to stand for one hour at least. The medusa will sink through

the diffusion gradient between the alcohol and oil. Pipette away all the alcohol and transfer medusa to new oil

(c) In the actual mounting place some Canada balsam on a slide and allow it to air dry slightly. Put the medusa on to this, cover with more balsam and allow this to harden a little. Place more balsam on and add a cover-glass, which will not now crush the medusa.

*Ascaris*.—Specimens should be kept in 2 per cent. formalin or 70 per cent. alcohol in large flat jars. On arrival from an abbatoir, they should be carefully washed and the two sexes sorted out. Broken specimens should be discarded; all the rest should be treated as carefully as possible to avoid injury and excessive curling. Sections are not easy to prepare, but they can be cut in paraffin wax if the cuticle is first treated with 5 per cent. KOH for several hours. This roughens the cuticle and also makes it more permeable to wax. Stain in Mallory.

*Proleptus*.—The dogfish usually provides specimens of a nematode, *Proleptus sp.* Pupils should make a mount of this as follows:

Place the specimen in the following mixture:

100 per cent alcohol	.	.	.	22 c.c.
Chloroform	.	.	.	15 "
Glacial acetic acid	.	.	.	5 "
Phenol crystals to raise volume by	.	.	.	10 "

To this add, drop by drop, oil of wintergreen, and then slowly add Canada balsam and finally mount the specimen.

*Tænia* (Tapeworm).—Living material should be fixed in Bouin's fluid, 70 per cent. alcohol or mercuric chloride. "Whole mounts" should be made after very prolonged overstaining in Borax carmine. Sections should be stained in hæmatoxylin or Mallory. Series should be made to show different stages in "ripeness" of the proglottides.

*Distomum* (Liver Fluke).—If living specimens are obtained, they should be pressed between two slides and fixed in 70 per cent. alcohol, etc. The excretory and



alimentary systems may be injected with a suitable dilute medium, using a very fine pipette drawn out of hard glass. The injection is made through the body wall at the posterior end. The animal should then be pressed between slides and hardened in 90 per cent. alcohol and finally cleared and mounted.

Whole mounts of stained specimens should be made; overstain for very long periods and gradually differentiate. Serial sections will be required which should be stained in Mallory or hæmatoxylin.

*Lumbricus* (Earthworm).—Kill by chloroform vapour. Dropping into boiling water may be tried. Dissect immediately after death in warm normal salt solution. The nephridia show up well in a demonstration dissection after running saturated aqueous mercuric chloride over the dissection. A demonstration of the blood-vessels can be made by opening up the worm and leaving covered by aqua regia for two hours. The blood-vessels are black on a yellow ground.

A series of "transparencies" kept in tubes are useful. They are prepared by taking small worms and killing them in 70 per cent alcohol, staining in Borax carmine and clearing in cedar oil. Some should be cleared but unstained. Various regions should be attempted, such as :

- (a) Complete anterior end.
- (b) Complete anterior end cut sagittally
- (c) Complete segment of posterior end
- (d) The ovarian segment with ovaries.
- (e) Ventral wall to show nerve cord, vas deferens, etc.

Examine with a binocular microscope if possible, using both transmitted and reflected light.

*Hirudo* (Leech).—Transverse serial sections may be prepared as follows: The leech should be anæsthetized with menthol, only a crystal or two should be placed on the water surface. When immobile, it should be fixed for about eighteen hours in Bouin's solution. The sections should be stained in Mallory or in Delafield's Haematoxylin; if possible, have series in both stains.

*Periplaneta* (Cockroach).—Microscopic preparations should include .

1. Mouth parts. These must be carefully dissected away one by one from the ventral surface of the head which has been severed from the body and which is pinned through the occipital foramen. The mouth parts should then be gently boiled in 2 per cent. KOH for five or ten minutes, or left in the cold solution overnight. This will help to depigment them and also remove all traces of flesh. Mouth parts are mounted all together unstained on one slide.

In making preparations of mouth parts, it is a mistake to continue the potassium hydroxide treatment for too long because, with complete transparency, it is impossible to see the limits of the sclerites.

2 Salivary glands. These must be carefully removed below water and floated out on to a cover-glass. On this they are carefully spread out and fixed in 70 per cent. alcohol, and subsequently stained and mounted. All operations are carried out with the cover-glass bearing the glands, since without such support they would collapse. Dehydration must be very carefully and thoroughly carried out.

3. Tracheæ. Pieces of trachea should be dissected out and mounted in glycerine jelly and in Canada balsam. They can be unstained or stained in 1 per cent. methylene blue, or stained by silver impregnation methods. It is stated that by injecting *Indigo white* into the body cavity and then plunging into air-free hot water, the tracheæ are shown up in blue colour.

Any of the above notes can be applied to other insects, such as the bee or wasp. The pupil should prepare a series of comparative slides of mouth parts. In the case of small and delicate insects, such as gnats and mosquitoes, the whole head can be mounted.

*Asterias* (Starfish).—If living specimens are obtained, they must be preserved in 5 per cent. formalin, the dorsal surface of each arm should first be opened to

allow of penetration of the formalin. Microscopic sections should be cut at about  $10\mu$  and stained in Mallory's Triple Stain or in Borax carmine. They should be obtained from a small and young starfish, which must be decalcified in 1 per cent. hydrochloric acid in 70 per cent alcohol. The regions required :

T.S. Arm near to disc.

T.S. Arm at extreme tip.

T.S. Across disc on various radii

Horizontal sections through the disc are often of value.

Whole mounts of the larval form should be purchased, or tubes of the larvæ purchased and stained in Light Green and mounted in balsam.

## AQUARIA

### 19. NOTES ON THE MANAGEMENT OF AQUARIA

*Ian T. Hamilton*

When collecting living material, the greatest danger of enthusiasm is over-collecting. All aquatic life requires considerable spacing out, owing to the small amounts of oxygen that are present in the water compared with the amount in an equal volume of air. In a series of small aquaria, there is always the danger of overstocking in order to create an impression, but one should be content with a small number of inhabitants in each tank, irrespective of any impression it is wished to create. Life in a small aquarium is very unnatural, and small changes, particularly changes of temperature, are rapidly transmitted through the whole : because of such changes it is not always possible to compare the results and methods of a large aquarium with those of a small one. For this reason, it is believed that these notes may supplement the information that is supplied in certain aquarium books.

Artificial sea water can be made, but it is an almost

worthless substitute for real sea water, which contains large amounts of microscopic life, called plankton, upon which many animals feed, together with much organic life, known as the nannoplankton, which is invisible under the ordinary microscope. Artificial sea water contains neither plankton nor nannoplankton, and therefore, though chemically equivalent, lacks the necessary "life giving" qualities.

From the point of view of teaching biology in schools, the sea-water aquarium, though more expensive, is far more valuable and instructive than a fresh-water aquarium. A greater range of animal life can be kept in sea water, while the fauna of fresh water is practically restricted to fishes, mussels and crayfish. Sometimes the aquarium can be used as a storehouse for material for teaching, and when one lives far from the sea, this is clearly an advantage. Fresh-water life, on the other hand, often requires no storing, for it can be collected locally; furthermore, most freshwater fauna is too small to be kept in an aquarium tank and can be kept alive in jars through which air is bubbled.

*The General Management of Aquaria.*—This involves obtaining food for the animals, removing uneaten food and dead animals, keeping a check upon the supply of water to all tanks and keeping records of interesting events.

Food consists mainly of chopped worms, and in frosty seasons, when worms are scarce, this diet may be replaced by small pieces of meat, though living food is preferable. The diet may sometimes be varied with *Gammarus* and Water Fleas, but worms can be used at all seasons for all animals. Food should be supplied to all tanks about three times a week.

When a tank is cleaned, care should be taken that no sand is rubbed against the glass, in order to avoid scratches. It is preferable to allow the sides of the tank to remain dirty, for there is a considerable amount of natural algal growth which, if disturbed, always upsets

the balance that is being made in the tanks. If aquatic plants are present, it is as well to fix a collar of lead around their bases; otherwise they will float to the surface and eventually block the exit pipe of the tank. *Elodea* and *Vallisneria* are suitable for fresh-water aquaria. For marine aquaria, seaweed should be frequently renewed.

If iron pipes are used (they have the advantage of cheapness and purity), the water should flow through them as quickly as possible, to dislodge the rust that tends to collect inside. In hot weather, water should always be supplied at the maximum rate, to reduce warming of the water to a minimum.

A pipe that goes to the bottom of a tank appears to provide better aeration than that produced by a jet. Bubbles of air can be introduced into the inlet pipe of any tank by having a short connection of *perished* rubber tubing. If the tank can be drained at the bottom, for cleaning or other purposes, the hole and stopper should be dried and both given a generous coating of vaseline. The prevention of leaks requires a great deal of care and attention.

It is necessary that all results, negative or positive, should be kept in a Log Book, to avoid the repetition of mixing the wrong animals together; this will also help considerably when making up an order for new stock. On the whole, it is advisable to keep carnivorous forms rather than sedentary and filter-feeding forms.

*The Stocking of Aquaria.*—(a) *General Note.*—In ordering material for stocking aquaria, it is advisable to plan out in advance the distribution of the stock that is to be ordered. Certain animals cannot be kept in the same tank as others, and thus it is desirable that each tank, as far as possible, be kept for a special purpose.

(b) *Marine Worms.*—Provided that sufficient worms are available, these can be displayed satisfactorily. Tube worms should be planted in a bed of sand and the rest of the tank covered with small gravel so that errant

forms can find the necessary shelter yet remain partly visible. Species which can be cultivated include *Sabella pavonina* and *Nereis spp*

(c) *Echinoderms* —The Echinoderm tank requires a considerable amount of fairly coarse gravel, to which the tube feet of the animals can adhere. Species which have been found to remain alive for several weeks are *Asterias rubens*, *Ophiocoma nagra*, *Cucumaria spp*, *Holothuria nigra* and *Synapta spp*. The small sea urchin, *Echinus miliaris*, may be kept alive for a few weeks in a small aerated pot, but not in the aquarium.

Starfish may be introduced into the tank, and should be held against the side of the tank so that they can begin life in their new environment with a firm foothold. Starfish feed on mussels, and about the same number of mussels as starfish will be required; dead mussels must not be left in the tank.

(d) *Anemones*.—Anemones should be kept in a tank where the water rises and falls more or less like the tides. See No. 22, p. 71. They can be fed with chopped worms about three times a week, and must be fed individually because of their anchored habit; stirring the water before feeding usually stimulates them to open, but at Plymouth and the Zoo Aquarium water saturated with chopped fish or squid is used for this purpose. Small crabs, about 1 in. broad, can be kept with the anemones and form another source of food; larger crabs should not be included since they attack and dislodge the anemones.

To enhance the appearance of the tank, which otherwise shows little movement, Wrasse (*Labrus maculatus*) can be introduced, though occasionally one may be caught by the anemones. Anemones which may be reared include *Actinea equina*, *Sugartia bellis*, *Thoe spp*. and *Actinobola dianthus*.

(e) *Marine Crustacea* —Shore crabs (*Carcinus mœnas*) may be kept easily but must be well fed; otherwise they will kill and eat one another. The edible crab (*Cancer pagurus*) will not live in these tanks. Unless the

crabs are small, they should be kept in a tank by themselves. An allowance of about 54 sq. in. of ground space per crab is a minimum for these animals. Burrowing crabs, *Conoplax rhomboides* and *Corystes cassivelaunus*, do not acclimatize themselves at all well, and *Portunus depurator*, a quite common swimming crab, is not worth keeping, the mortality being very high.

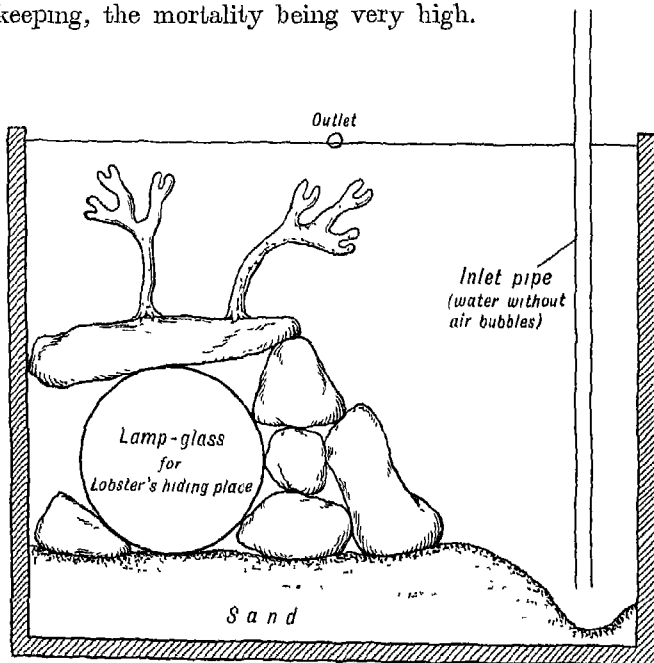


FIG. 12.—Arrangement of Tank for keeping Lobster.

Small lobsters have presented a difficulty in the past, but it is found that if they are not supplied with the same amount of aeration as in other aquaria tanks, they will remain alive for as long as nine months. It is well to keep one lobster at a time; two in the same tank will fight, but small crabs may be kept with this animal. These provide food, and if a crab is chased by a lobster, the habits of the latter may be observed; normally it retires to a hiding-place and is rarely seen.

The arrangement of a tank for keeping a lobster is illustrated. The quantity of sand should be small; otherwise the animal will burrow into it and make hillocks of sand in front of its shelter. A fatal period is usually the time when the animal is undergoing ecdysis. In fact, this is true of all Crustacea that may be kept.

The hermit crabs, *Eupagurus Bernhardus* and *E. Prideauxii*, with their respective commensal anemones, *Sagartia parasitica* and *Adamsia palliata*, may be introduced into this tank, and will adapt themselves to their new surroundings; if the anemones become dislodged, however, their chance of survival is small.

(f) *Marine Fish*.—Marine fishes that may be kept are Top-pot Blennies (*Gattorugine*), *Cottus*, Mullet and most rock pool fish. The Rockling (*Motella sp.*) seems to suffer considerably from attacks of the Tape worm, *Gyrodactylus*, and there is always a risk of this infection spreading to other fish. It can be recognized by the appearance (often over the eye) of white patches on the dark brown surface of the Rockling. No cure has been found, and it is best to remove and kill the infected animal as soon as the disease appears.

(g) *Marine Mollusca*.—Nudibranch molluscs will sometimes remain alive, especially if seaweed is placed in the tank; this must be replaced frequently or the water will become foul. *Doris* has spawned in these tanks. Gastropods can be kept fairly well, but since the shore forms are able to live out of water, they require watching or they will crawl out of the tanks and disappear. *Buccinum* will spawn and live quite well.

(h) *Fresh-water Fish*.—A number of varieties of these can be stocked, such as Sunfish, Bitterling, Carp, Dace, Perch, Minnows, Goldfish, varieties of Tench, Orfe and Trout. Trout require a plentiful supply of water, but will live a long time when once established. When they reach about 7 in. in length, the top of the tank should be covered with a zinc gauze frame, since they may possibly jump out of the water.



(i) *Crayfish*.—Crayfish may be kept, but the same remark about ecdysis applies here as to the Lobster. Like all Crustacea that are supplied "dry" by dealers, they should be gradually immersed in water on arrival, to replace the air that is filling the spaces under the carapace.

*Reproduction in Aquaria*.—This is a process that delights all keepers of aquaria, but it is not until the appearance of a post-larval new generation that it is possible to see the process finished. Spawning may take place with *Doris*, *Buccinum*, *Carcinus*, Crayfish and Axolotl. One cannot be certain with aquatic forms that fertilization has been effected, and this may account for many failures, but even when one is fairly certain that this process has taken place, the eggs do not mature. Larval forms, if produced, may escape into the surrounding water, where they soon become the victims of some other inhabitant.

Axolotl tadpoles have been kept for about three weeks, and although they were isolated, carefully fed upon *Cladocera*, supplied with running water and their tank cleaned, none grew larger than about an inch before dying.

Four axolotls have been kept for several years. Twice they have spawned and at no particular season. The process has not been observed and probably takes place at night. Eggs laid in the summer of 1933 hatched in nineteen days, but those laid in February, 1934, took as long as six weeks to hatch. These animals occasionally bite off parts of one another's limbs, but these are soon replaced, and in one specimen a double hand has now been produced by regeneration.

## 20. SMALL AQUARIUM AERATOR

W. B. Barker

The apparatus shown in Fig. 13 can be used to aerate a small aquarium and requires but a small consumption

of water. A quick succession of drops of water entering the bottle A (about 1 pint capacity) displaces an equal volume of air through the water in the aquarium. When the bottle is full it is automatically emptied by the siphon, fresh air being admitted through the Bunsen valve B.

The following should be closely adhered to in constructing the apparatus :

1. The siphon tube must not be greater in internal diam. than  $\frac{1}{4}$  in (the ordinary glass tubing used in chemistry lab will do), or the siphon will not function. The longer limb of the siphon should be as deep as conveniently possible to increase the rate of siphoning.

2. A wide tube (about  $\frac{3}{4}$  in. internal diam.) must be sealed on to the end of the shorter limb of the siphon inside the bottle, or the siphon will not break when the bottle has been emptied.

3. The actual height of the tube C and of the siphon will vary with the depth of the tube immersed in the water in the aquarium, but in any case must be an inch or so greater.

4. It is advisable to cut the V-slit in the rubber tube of the Bunsen valve with a razor blade and to dust the cut edges with French chalk, to avoid sticking.

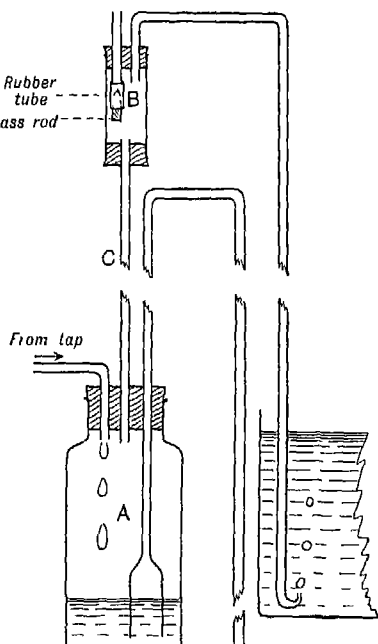
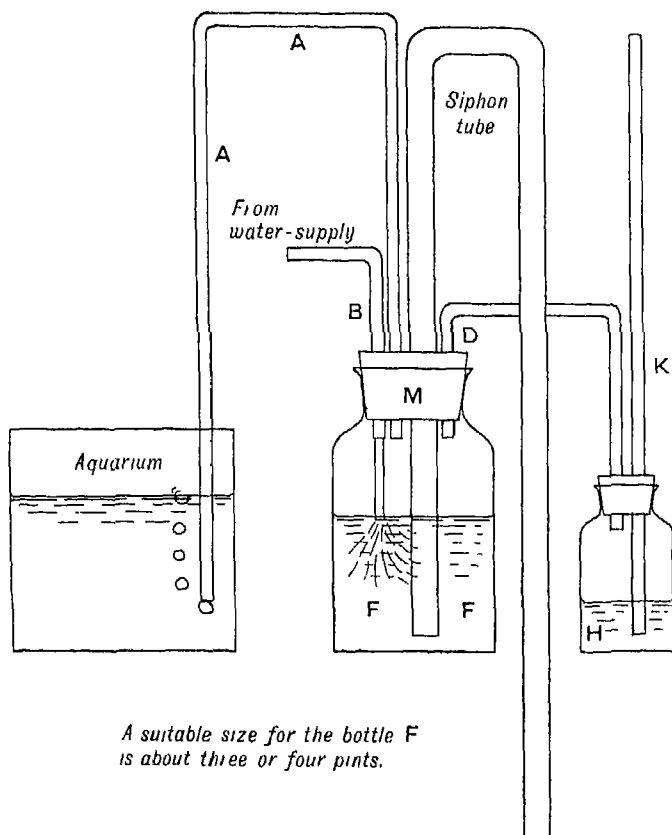


FIG. 13.

## 21. AERATING APPARATUS FOR AQUARIA

*Maurice J. Glenn*

The aerator is designed for use with a water supply and requires only a slender constant stream. Although the method of working is obvious from Fig. 14, there are a few precautions to note in order to ensure reliable aeration.



*A suitable size for the bottle F  
is about three or four pints.*

FIG. 14.

1. The siphon tube must be wide (about  $\frac{3}{8}$  in.), since the outgoing stream is required to race the incoming tap

water. Again, a wide siphon reduces the interval between successive bubbleings. If the siphon is narrow, premature siphoning may occur since water remains in the longer limb and drags more over from F.

2 The outgoing tube A, the siphon and the air intake K must all be at least 1 ft. above the top of the bottle F. The back pressure—when air is being forced over—of water in A from the aquarium is enough to spill water from K or to start the siphon should either be short. A must be long to prevent tap water leaking into the aquarium when F is full.

3. The stream of water from B should be regulated so that it runs at about one-half or one-third of the rate at which the siphon empties

4. The bung M must be of rubber and each tube should be a tight fit.

5. The tube A must be fairly wide and not drawn out at its tip. A narrow opening causes a great increase in resistance to the air, and water will probably be forced out of K or over the siphon.

It will be seen that the air trap H could be replaced by an open tube through M, dipping to the bottom of F. This is even simpler, but it has been found that when the siphon starts, its speed is retarded by a pressure equal to the height of the water column in F. The trap H is extremely simple and is worth the slight extra trouble.

## 22. TIDAL TANK

*Ian T. Hamilton*

The rise and fall of the tide can be produced in an aquarium tank by constructing the following simple device. About 2 in. below the normal overflow, a hole is bored to allow a piece of glass tubing to pass. The diagram is self-explanatory. As water rises in the tank, it enters the hole A, and rises in the tube until it has reached the level E, whereupon the water drops to G,

forming a siphon. The water level then falls again, and somewhere between A and B the siphon is broken. The side piece, C, is attached in case the opening, A, should

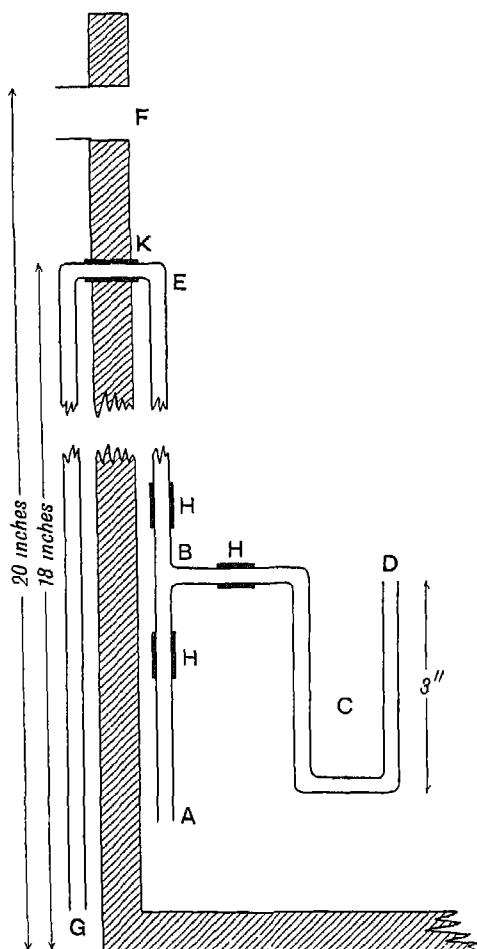


FIG 15.

become blocked with sand. As soon as the tank fills to the level D, water enters the U-tube, and the tube BE begins to fill as the level rises.

It is necessary that the *normal* outlet should also be available, for should the siphon fail, the water will rise to F and will run out of the tank in the usual manner.

The "tide" can be made to rise twice a day by regulating the inflow so that water empties twice as fast as it comes in.

## BLOOD SYSTEMS

### 23. INJECTION OF ANIMAL BLOOD SYSTEMS

*T. L. Green*

*A Simple Injection Mass* (Guyer).—Grind up 5 gm. of carmine in 10–12 c.c. water and add drops of ammonium hydrate until the solution becomes transparent. Soak 50 gm. of best sheet gelatine in water, finally melt it at about 40° C. and pour in the colouring matter. While it is cooling, add drops of 25 per cent. acetic acid till the mass becomes opaque and the smell is faintly acid.

If a blue colour is desired, make the gelatine solution and add an aqueous solution of Berlin blue.

*Scyllium* (Dogfish).—Injection of the blood systems can be prepared with ease if fresh material is obtainable. Perform the injection with a brass syringe, hypodermic needle or by rigging up a constant pressure device with jars, tubes and a glass cannula. The injection mass described later should be used.

(a) For the central arterial system, inject through the conus of the heart.

(b) For the dorsal system, cut off the tail end behind pelvis and inject through dorsal aorta.

(c) For the venous sinuses, inject through the openings into the cuvierian ducts and sinus venosus.

Before injecting the coloured mass, inject a warm 1 per cent. solution of sodium citrate or 0.5 per cent. salt solution to clear the vessels.

*Lepus* (Rabbit) and *Columba* (Pigeon).—The instruments

and injection mass described for the dogfish can be used for the rabbit.

The injection should be carried out at once after death. The femoral artery and vein should be severed on one side to allow bleeding to drain the systems.

The locality chosen varies with the needs, but generally injections are made through the heart and great vessels, the hepatic and portal veins, the dorsal aorta and the femoral vein or artery.

Warm salt solution (0.7 per cent.) or a vaso-dilator (sodium citrate) should be injected first.

*Astacus* (Crayfish).—The vascular system should be injected by holding the animal below the surface in a bowl of warm water (just above room temperature), and cutting away the carapace dorsally. Inject with a fine pipette through an ostium, using a coloured gelatine mass. Plunge at once into boiling water, and, when dead, place in cold water.

## CULTURE MEDIA AND CULTURES

### 24. PREPARATION OF MEDIA FOR THE CULTURE OF BACTERIA

*L. W. White*

*Nutrient Agar* (for general use).—Place 1,000 c.c. of water in a flask of about 1½ litres capacity. Add 10 gm. of Peptone, 3 gm. of beef extract (e.g. Bovril), 5 gm. of common salt (unless the beef extract is salted). Thoroughly wet 15 gm. of agar agar and add this to the contents of the flask. Heat in the steam sterilizer until the agar has dissolved. Make the medium faintly alkaline to litmus by adding sodium carbonate. Allow to cool somewhat and add one egg beaten up (including the shell); mix well and heat again for a short time. This will "clear" the medium, which must then be filtered through special paper for agar. Cut most of the stem off a large funnel and stand the funnel in a large beaker.

Place this in the steam sterilizer and keep the water hot until filtering is completed (probably all day at least). Transfer the filtered medium to a flask, plug the neck with cotton-wool and sterilize in the steamer on three successive days, the water being kept boiling, on each occasion, for not less than half an hour after all the medium has melted.

*Asparagin Agar* (for soil bacteria).—Prepare 1,000 c.c. of an ordinary water culture solution ("complete solution") and add to it 0.5 gm. of asparagin and 15 gm. of agar agar. Thoroughly wet the agar agar before adding it. Sterilize the medium on three successive days as for nutrient agar.

## 25. CULTURE OF SOIL MICRO-FUNGI

*E. J. Moore*

The micro-fungi of the soil form an interesting field for investigation, as not only do many of them illustrate typical fungal development, but there is always the possibility of the discovery of new types.

The culture of the fungus flora of a particular soil can be carried out as follows. Boil a number of Cress seeds for twenty minutes in order to sterilize them and to cause the mucilaginous testa to swell; place a layer of soil in a Petri-dish, cover with  $\frac{1}{2}$  in. water and drop in the seeds; after two or three days transfer the seeds carefully to another sterile dish, cover with not more than  $\frac{1}{2}$  in. water, and leave the culture to develop.

Seeds can be readily examined microscopically at intervals, the hyaline mucilage forming an excellent culture medium for such a purpose.

## 26. CULTURE OF "LAMINARIA" GAMETOPHYTES

*E. J. Moore*

The gametophyte of *Laminaria* is more often studied from a textbook figure than from actual specimens, though



these are fairly easy to grow, even in a laboratory not close to the sea.

Fronds of the seaweed, bearing ripe zoosporangia, should be obtained during the winter or spring, cut into suitable pieces and placed in a shallow glass dish containing sea-water. Both the material and the sea-water can be obtained by post so long as they are fresh when packed and transported quickly.

Several glass slides or cover-glasses should be placed in the dish in order that growth shall occur on them. After a day or so, to allow of the liberation of the zoospores, the pieces of frond should be removed with wooden forceps or a glass rod (*in no circumstances with metal*), and the culture gently aerated once a day with a fountain-pen filler.

Slides or cover-glasses can be removed and examined at intervals for stages of development.

## HERBARIUM

### 27. PRESERVATION OF PLANT SPECIMENS

*E. H. Ellis*

#### *Preparation of Pressed Plants for Herbaria.*

(a) *Ordinary Land Plants*.—Arrange the specimens carefully on a sheet of absorbent paper, such as “grey filter paper,” separating any overlapping parts with strips of the same material; cover with another sheet of absorbent paper and press, either by means of a board and a light weight, or by boards above and below, held together by webbing straps.

If several specimens are being dried at the same time, sheets of corrugated paper should be inserted at intervals in the bundle. Drying-papers should be changed daily until the plants are completely dry.

(b) *Succulent Plants*.—These must first be killed by

immersing them in boiling water for a short time, and then pressed as before.

(c) *Conifers*.—Before pressing, plunge the specimen into a 10 per cent. solution of Formalin for ten minutes, or place it in a closed chamber containing Formol vapour, for two hours. This treatment should prevent excessive defoliation, but if this still occurs, the specimen should be sprayed with a solution of cellulose acetate in acetone, of a suitable consistency.

(d) *Aquatic Plants*.—Place the specimen in a shallow dish containing sufficient water for it to float naturally; introduce a sheet of thin paper into the dish, remove paper and specimen carefully, drain off the surplus water, and press between absorbent paper.

(e) *Seaweeds*.—Use the method given for Aquatic Plants, but replace the fresh water with sea water, or a solution of 35 gm. of Tidman's Sea Salt in 1 litre of water.

(f) "*Slime Fungi*" and *Lichens*—Some lichens may be pressed normally, but crustaceous forms and Slime Fungi should be allowed to dry slowly in air, without pressing, and mounted in small boxes.

### *Mounting Dried Pressed Specimens*

Dried specimens should be fastened to suitable sheets of cartridge paper by a mixture of gum tragacanth and gum senegal (best grade Gum Arabic); this can be reinforced, if necessary, by strips of linen tape over stems, petioles and flower stalks.

### *Storage of Dried Pressed Specimens.*

To prevent insect attacks, herbarium sheets should be stored in a cabinet containing a few flakes of naphthalene or crystals of para-dichlorbenzene.

Certain specimens, such as Willows and Composites are best treated by painting them with the following solution · Mercuric Chloride, 50 gr. , Phenol, 50 gr. ; 94 per cent. Alcohol, 1,000 gr.

*Preserving Specimens to Retain the Colour.*

(a) *Drying with Sand.*—Fill a tin tray of suitable dimensions with fine dry silver sand ; place it in an oven to heat at about 200° F., agitating the sand occasionally to ensure a uniform temperature throughout. When heated, remove sufficient sand to accommodate the specimen, which is laid in the warm sand in the tray and the remaining sand poured over it carefully by means of a funnel. The tray is now replaced in the oven and the heat turned off ; when the sand is completely cool, remove the tray and take out the specimen, which can be mounted on a card in a glass-fronted case.

(b) *Preserving in Liquid* —To preserve the green colour of specimens, boil them gently in a glazed earthenware vessel containing the following solution : Copper Acetate, 25 gm. ; Glacial Acetic Acid, 100 c.c. ; Water to 1 litre.

The length of time varies according to the nature of the specimen, ordinary mesophytes requiring about five minutes, but plants with a resistant epidermis may require half an hour or more.

After boiling, remove the specimen with wooden forceps, rinse it carefully in water, and preserve in a suitable museum jar containing a 4 per cent. solution of formaldehyde.

The jar should be sealed with seccotine or bichromated gelatine.

(c) *Sealing Mixture for Museum Jars.*—Bichromated Gelatine is an efficient cement for jars containing liquid, since the gelatine is made insoluble by light.

To prepare the mixture, dissolve 25 gm. of gelatine, Nelson's No. 1, in 1,000 c.c. of a saturated solution of potassium bichromate, and store it in collapsible tubes.

(Metal tubes are sold with the lower end open ; these are easily filled and the end sealed by pressing together, turning over twice and pressing together with pliers.)

For use, the tube should stand in warm water until the gelatine is fluid ; a sufficient quantity is squeezed out

round the rim of the jar, the glass cover placed on the jar, and retained with pressure until the cement sets.

(d) *Preserving the Colour of Green Algae*.—Store the material (fresh water or marine) in lactophenol containing a small quantity of copper acetate. Lactophenol is prepared from the following :

Phenol crystals	20 gm.
Lactic acid .	20 „
Glycerine .	40 „
Distilled water	20 „

## PLANT ANATOMY

### 28. XEROMORPHIC CHARACTERS

*Eric Ashby*

A survey of the commoner xeromorphic characters may be made from a study of the following types .

*Sunken Stomata*.—Cut transverse sections of leaves of *Aloe* (Liliaceæ), *Dasyliiron* (Liliaceæ) and *Hakea suaveolens* (Proteaceæ). Stain in safranin and hæmatoxylin.<sup>1</sup> Safranin will stain all lignified tissue, hæmatoxylin the cellulose and cell contents, and the cuticle will remain unstained.

Note the stomata protected by “ vestibules ” of cuticle, and the development of the mechanical tissue.

*Protected Stomata*.—Cut transverse and longitudinal sections of leaves of *Erica* (Ericaceæ) and *Empetrum* (Empetraceæ) (species immaterial), and *Nerium Oleander* (Apocynaceæ). Stain as before. Note the orientation of the stomata in *Erica* and *Empetrum* ; the stomata are sunken in grooves in these two plants and in pits in *Nerium*. The function of the glands in *Erica* and *Empetrum* is not known.

<sup>1</sup> Safranin 50 per cent alcoholic, and hæmatoxylin 50 per cent. alcoholic solutions. Stain in safranin first for half an hour, wash in methylated spirit, stain in hæmatoxylin for about two minutes, wash again. Dehydrate with alcohol, clear with clove oil and mount in Canada balsam.

*Succulence*—Cut sections of the leaves of *Roechea falcata* (Crassulaceæ) Note the protection given to the stomata by the peltate epidermal cells, and the large size of the sub-epidermal cells, and the paucity of the lignified tissue.

*Induced Xeromorphy in Sinapis alba.*—Sow White Mustard, *Sinapis alba*, in ten pots, filled with soil. Water five of these pots frequently, taking care not to add an excess of water at the beginning. Water the other five pots normally until the first foliage leaf begins to unfold. After this, give them as little water as possible to prevent their actually wilting. The dry culture will develop more slowly than the damp culture and some of the “dry” plants will probably die. When from five to six leaves are produced, the plants from the two cultures are collected (including the root systems), washed, and preserved in 70 per cent. alcohol. The anatomy of the leaves from the same tier of “damp” and “dry” plants is then compared. The leaves are prepared for examination as follows :

(i) For the estimate of the relative thickness of cuticle, stain transverse sections of the leaf for 20 minutes in a 5 per cent. alcoholic solution of Scharlach Red.<sup>1</sup> Note the difference in thickness between the cuticles of “damp” and “dry” leaves, the former should be about half the thickness of the latter.

(ii) For measuring cell size and the number of stomata per unit area, cut surface sections of the abaxial (lower) epidermis. The sections will usually be wedge-shaped, and at the thin end the stomatal frequency can be measured. Stain the sections in iodine and observe under a  $\frac{1}{6}$ -in objective.

(iii) The increase in conducting tissue in the leaves grown under dry conditions may be demonstrated by the following method. Stand the leaf for twenty-four to forty-eight hours in freshly prepared eau-de-Javelle. By

<sup>1</sup> Other stains, e.g. Sudan III and osmic acid may be used for staining the cuticle.

this time the leaf should be bleached and cleared. Wash for twenty-four hours in running water. This is best done by putting the leaf in a small beaker covered with muslin, under a slowly running tap. After washing, leave the leaf in ammoniacal fuchsin for twenty-four hours. This stain is prepared by adding 5 per cent. basic fuchsin to 0.88 ammonia until a permanent straw colour is produced. The stain is then filtered and ready for use. After twenty-four hours in ammoniacal fuchsin, the leaf is washed in methylated spirit. In the spirit the lignified tissue will stain deep red, and the unlignified tissue remain clear and colourless. The whole leaf may be dehydrated and mounted in Canada balsam. It will be easy to observe and measure the vascular system of the leaf

*Gradient of Xeromorphic Characters in Ipomœa.*—In many plants there is a progressive increase of xeromorphic characters from the lower leaves to the upper. To investigate this gradient, grow *Ipomœa purpurea* (Convolvulacæ) in a cold greenhouse. The plants will grow rapidly and must be trained along a pole. Suppress all the lateral buds before they sprout. When the plant is about 12 ft long, the successive leaves may be collected and the cell size of the lower epidermis and the number of stomata per unit area may be measured as described in the previous experiment

That the response is correlated with the water supply may be demonstrated by growing *Ipomœa* plants side by side, watering one set of plants freely, and giving the other set the minimum amount of water necessary to keep them alive. The decrease in cell size of successive leaves will be much more pronounced in the dry cultures.

*Transpiration Rate of Successive Ipomœa Leaves*—The differences in cell size obtained in the last experiment result in differences in stomatal frequency, which, in turn, cause differences in the transpiration rates of successive leaves. This can be demonstrated by cutting off leaves

<sup>1</sup> For particulars of the significance of the cell size, see Ashby, *School Science Review*.

under water and measuring their water loss from a Thoday potometer<sup>1</sup> Differences as great as 30 per cent. in the transpiration of lower and upper leaves can often be obtained.

### RESPIRATION

#### 29. RATE OF INTAKE OF OXYGEN IN RESPIRATION

##### *D. Thoday*

Fig. 16 shows the apparatus spread out diagrammatically. A is the experimental chamber, B a compensating chamber similar to it. A and B are connected by a manometer C, containing a light (non-volatile) oil. D is a graduated tube and E a reservoir, containing water, which can be raised or lowered. Equal volumes of dilute potash (say 10 per cent.) are introduced carefully with a pipette into the bottom of each vessel. The respiring material is suspended from a hook in the rubber stopper of A

With the outlets in A and B open, E is lowered till the level in D is conveniently near the lowest graduation, and A and B are then tightly closed. By fine adjustment of E, the oil is brought to the same level in the two arms of the manometer, and the first reading taken in D.

Provided that all joints are airtight and the temperature is the same in A and B, whenever the oil level is adjusted to equality in C, changes of volume due to changes of temperature or pressure are compensated and the volume read off in D will be accurately comparable with the initial volume.

The two outlets to A make it possible to change the air without disturbing the apparatus, by drawing air through with an aspirator or blowing it through with a bicycle pump or spray bulb.

The apparatus can be set up as in Fig 16, supported on

<sup>1</sup> See No 31, p 86

ordinary retort stands, at *a*, *b*, *c*, and *E*. The more compact arrangement illustrated in Figs. 17, 18, and 19 is, however, more convenient, and can be kept as a piece of permanent equipment; the whole apparatus can be

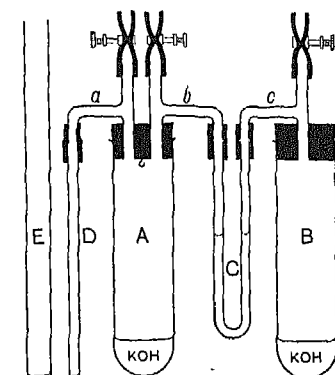


FIG. 16

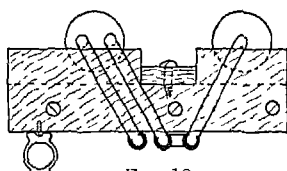


FIG. 18

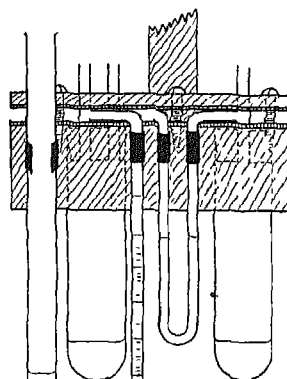


FIG. 17

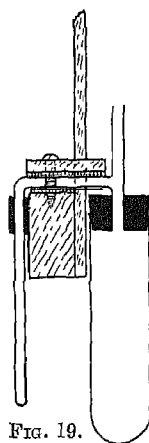


FIG. 19.

immersed in water, which keeps the temperature steadier and more exactly the same in A and B. For this purpose the whole apparatus is fastened to a supporting block of wood, with A and B behind, C and D in front, E in front or at the side, as may be convenient. It has to be remem-



bered that A and B have to be removed from time to time so they must be clear of the block.

The apparatus is constructed of the following parts ·  
*a, b, c*, 3 T-tubes, with fairly long stems ;

D, a 5 c.c. graduated pipette (or burette), with the tapered tip cut off ;

E, a larger burette tube or similar ungraduated tube ;

C, bent from ordinary tubing ,

A and B, *strong* boiling tubes, or a pair of similar bottles.

In either case the rubber stoppers must be pliable and fit well. The hook in A may be a bent pin or a piece of wire or, better, can be made of glass. The three outlet tubes must be fitted with thick-walled rubber tubing and screw clips. Pliability here would lead to errors. (Glass stopcocks are, of course, an improvement.)

Some sources of error call for notice (1) The handling of A in inserting the respiring material warms it slightly. With thick glass it may be half an hour or more before the temperature of A is equal to that of B. (2) A possible source of error is the transfer of water vapour from D to the KOH solution, increasing its volume and producing an apparent increase of volume of the air in A. With dilute KOH this is small. A blank experiment will show whether it is appreciable. (3) Small errors of an irregular kind may arise from unequal wetting of the two arms of the manometer with oil above the oil level. This is particularly the case if the difference of level has been considerable before the reading is taken. In such a case it is necessary to over-adjust a little, so as to wet the other side, and then leave for a time to allow the oil to drain down. Both (2) and (3) are eliminated by the use of mercury in the manometer and in D and E, though the adjustment of levels is then less sensitive.

Marks on the two sides of the manometer, or an adjustable piece of celluloid attached to it, bearing a horizontal line (a scratch filled with glass pencil or crayon is good) are a great aid to accurate and consistent levelling.

The amount of material and the intervals of time should

be arranged so that the change of volume to be measured is at least about 0.5 c.c., and the air should be changed before the oxygen content drops more than a few per cent. below normal. A dozen soaked wheat grains on a small piece of wet sponge may be suggested for a first trial.

## SKELETONS

### 30. PREPARATION OF ANIMAL SKELETONS

*T. L. Green*

*Astacus* (Crayfish).—The exoskeleton should be studied in specimens from which the flesh has been removed. Carefully bisect a specimen longitudinally and lift away the viscera and musculature; then soak it in 2 per cent. KOH for two days. The remaining flesh should easily brush away; if not, replace in potash. The specimen should then be well washed in water and finally preserved in spirit, or dried and mounted on a sheet of cardboard.

*Scyllium* (Dogfish).—Skeletal material is prepared from fresh specimens, skinning them and then slowly simmering in water until the flesh is soft. Brush and pick it away, but do not allow the cleaning process to go so far that the elements fall apart. It is difficult to make a complete articulated skeleton. Preserve in 70 per cent. alcohol and glycerine, 7 and 3 parts respectively.

*Lepus* (Rabbit) and *Columba* (Pigeon).—Skin the animal, eviscerate, and cut away as much flesh as possible. Dis-joint into various regions—skull, fore- and hind-limbs, vertebral column, etc. Treat each part separately; in the case of young animals, the skull, and the feet. Do not carry the following processes too far or complete dis-articulation will result. Clear out as much as possible of the brain or it may swell and disrupt the skull.

Macerate the parts by any of the following methods:

1. *Cold Water Maceration*—Leave in cold water till flesh rots and can be easily removed. This method is offensive, but generally results in clean white bones.

2. *Hot Water Maceration*.—Simmer till flesh is easily removed. This method is quicker, but the bones are often discoloured.

3. *Chemical Maceration* (Holden).—Prepare two solutions :

(a)  $\frac{1}{2}$  oz. cresylic acid (= cresol,  $C_3H_4CH_4OH$  in commercial ammonia) and 6 gallons water.

(b) Hydrogen peroxide in water 1 per cent. strength.

Stew in (a) till tender, then remove flesh. Wash in water and simmer to remove ammonia. Next stew in (b) until bones are white and free of grease.

4. *Digestion Method* (Rowley).—Simmer gently in the following solution and remove flesh, then whiten in peroxide solution :

1 tablespoon sodium sulphide.

2 tablespoons pancreatin.

1 gallon water.

After any of the above methods, the bones should be well washed in cold water, soaked for a few minutes in 70 per cent. alcohol and then air-dried carefully.

It is very useful to have skulls sawn sagittally and transversely with a fret saw. The sutures may be marked with ink or the several bones painted with different colours.

Articulation may be carried out, using a small drill and fine wire. Teeth may be set in with dental wax or "durofix."

## TRANSPIRATION

### 31. PORTABLE, ADAPTABLE POTOMETER

#### *D. Thoday*

The essential feature of this potometer (Fig. 20) is the convenient disposal of the apparatus on a wooden stand with the shoot supported near the centre of the baseboard so that the whole apparatus is well balanced. The potometer tube is horizontal instead of vertical, with short vertical arms to take the shoot and a thistle funnel. The

horizontal stem of the potometer tube is bent to connect with a correspondingly bent capillary tube. Both are fixed to the baseboard with metal strips and screws (with rubber or cork packing). Under the straight part of the capillary is a millimeter scale, of varnished paper.

The particular form of stand illustrated is very easy to construct. The shoot and the funnel are tied securely

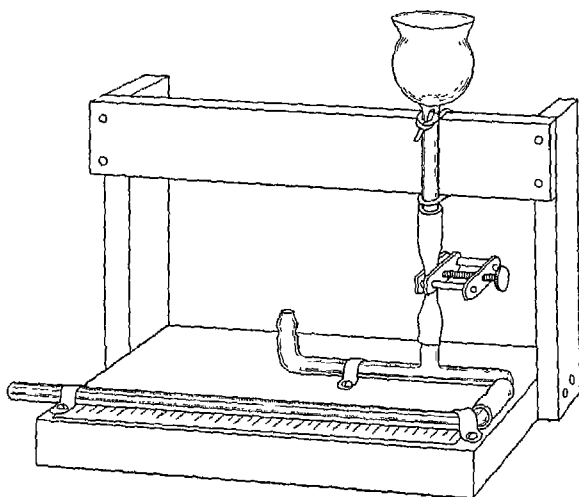


FIG. 20.

with string to the cross piece, which is high enough to allow manipulation of the rubber connections and the screw clip. Though the exact dimensions matter little, the following have proved convenient: baseboard 9 in.  $\times$  6 in.  $\times$   $\frac{3}{4}$  in.; uprights 6 in.  $\times$   $1\frac{1}{2}$  in.  $\times$   $\frac{1}{2}$  in.; capillary about 1 mm. bore; potometer tube of ordinary fairly stout tubing, or thicker walled (e.g. vacuum pump tubing) to withstand rough usage. A wide range of size of shoot is permissible. Smaller ones are connected directly; with thicker stems, a short, tapered glass connection is interposed, or the rubber-glass joint may be packed with a short length of narrow rubber tubing. The stem joint at least is, of course, wired.

In setting up the apparatus, the open end of the capillary is first closed with rubber tube and clip, and water run into the thistle funnel, with screw clip open, to fill both arms, including the rubber connection for the shoot. The shoot is then attached, the tube and clip removed from the end of the capillary, so that water flows through to fill the latter and the rest of the apparatus, and the screw clip closed.

If very large twigs (c.g. of evergreen shrubs in winter) are to be used, increased stability can be secured by extending the baseboard backwards (making it, say, 9 in. square, with the shoot quite central) or by weighting it.

## VIVARIUM

### 32. OUTDOOR VIVARIUM

*Ian T. Hamilton*

An outdoor vivarium should be erected in a sheltered but sunny spot, for even on the warmest of days, the inhabitants may prefer to hide in burrows rather than to make an appearance above ground. It is not advisable to build the vivarium near trees if other space is available, for though trees add an attractiveness to the surroundings, the falling leaves in the autumn collect in the water and make observation very difficult. Places where dust can be blown into the water should also be avoided. Probably the best surroundings will be found in the centre of a small grass plot.

*Construction.*—The vivarium about to be described has proved adequate in dimensions to accommodate as much material as is found in a locality with a rich fauna of Reptiles and Amphibians. It is D-shaped, but this was only a matter of choice. If space permits, it should be rectangular in shape rather than square so that one of the longer sides shall face the sun.

A pit, 13 ft. long, 9 ft. wide, was dug out to a depth of 2 ft. 9 in. and roughly levelled. Into this, 6 in. of coarse

rubble, consisting of odd stones and broken bricks, were placed and covered with a layer of fine rubble, the whole sloping down slightly towards one point, which was to

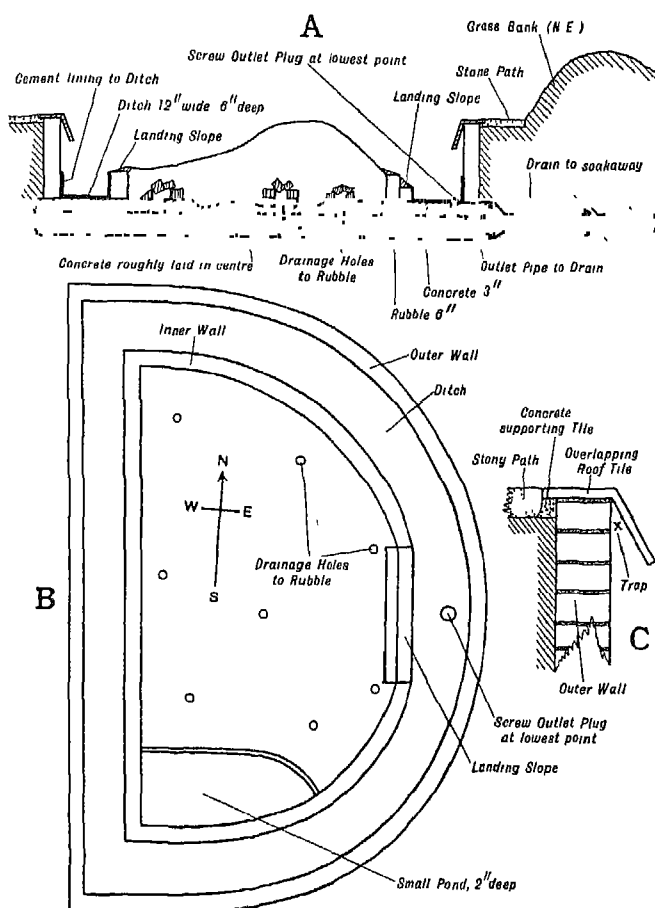


FIG. 21.

become the outlet. Up to this point it was possible for the work to be done by inexperienced boys. Over this was placed a layer of concrete, 3 in. deep and 2 ft round the margin. This required skilled labour in order to get the

general surface of the concrete level while allowing for a gentle slope towards the outlet hole already mentioned. These elaborate foundations were necessary because the vivarium was constructed in a sandy soil ; also, the ditch that was to surround the vivarium had to be kept water-tight and strong enough to withstand freezing in winter. Any sinking of the ground beneath would set up strains in this water-tight concrete.

Around the outside of this foundation, the outer wall, 8 courses high, was constructed in brick. Twelve inches inside this another wall of 2 courses was built ; the bricks can be laid by inexperienced boys. Inside this inner wall earth was placed. There was thus formed a ditch, 12 in. wide and 6 in. deep, which was then covered with a layer of waterproof cement (Pudlo). At the lowest point, a screw plug was let in flush with the bottom and connected with drain pipes to a soak-away. On top of the outer wall were placed some roof tiles, as shown in the figure. This has proved an effective trap for intending escapers. Recently, an underground tunnel has been constructed in the earth island in the middle, the tunnel being filled with dead leaves. This was made to allow animals to hibernate during the winter. The experiment has not proved entirely successful, for the inhabitants find in this tunnel a refuge from the view of the intending observer. The entrances of the tunnel have now been blocked up 6 in. from the mouths, and this appears to effect a compromise between the observer and the observed. The whole island is now covered with turf.

*Inhabitants.*—A number of animals have been kept inside this vivarium, both indigenous and foreign forms. Frogs and Toads may be collected locally, and these quickly seek out stones and pockets in the turf for hiding. Spawning has not taken place during the first year of construction, but tadpoles collected locally and kept in the surrounding ditch have developed legs and have grown into fair-sized frogs. Slow-worms may also be collected locally, and these burrow into the turf. If, however, they

get into the water, they are unable to climb out, and thus they want careful nursing. The Common Lizard may be collected locally, and Green Lizards and Wall Lizards may be bought from dealers. A number of those which have made their own burrows during the last winter (there were no winter quarters prepared at that time) reappeared again the following spring.

It is inadvisable to keep Grass Snakes in the vivarium, for they destroy some of the other inhabitants. Terrapins have been introduced, and after a short while become less shy and may be seen at most times in the day. During last winter, three specimens were left beneath 2 in. of ice in the ditch, and despite this, have made a satisfactory reappearance during the summer. These animals are carnivorous and appear to feed entirely upon tadpoles.

Edible Frogs have been introduced, but have not been kept long enough to show whether they are desirable or not. In transit, they often become badly damaged by jumping against the sides of the containing box and rubbing off pieces of their skins. They are retiring animals and at the approach of footsteps dive into the water; this, if not kept clear, causes them to become lost from view completely. Salamanders were introduced during the summer, but it is too early to decide whether it is advisable to keep these animals regularly in stock, for it is not yet known whether they will survive a winter out of doors. They may be stored in the vivarium for a week or two until wanted for class use. They do not like the water and hide in some damp shady pocket of turf. During the summer nights, they have been seen out of their hiding-places.

Aquatic forms of animal life can be introduced into the ditch, and water weeds with straggling stems can be conveniently kept there also. Though Burr-reed and Willow Herb will grow in the ditch, it has been found inadvisable to plant them, for they form a bridge from the vivarium to the outside, rendering escape an easy matter.

The disadvantage of turving the earth island is that when



the grass grows long, it is difficult to cut it without cutting in two the animals which burrow into the turf; furthermore, the cut grass blades fall into the ditch and make it untidy. If nothing is planted on this earth mound, it soon becomes untidy also. It is suggested that heather be planted to keep down the growth of weeds. The heather plants should not be tall, however, for again, they will form a bridge of escape. They can be confined to the centre of the island.

The water in the ditch does not evaporate very quickly, and only once in twelve months has it been replaced. A balance of plant and animal life seems to have established itself. The inhabitants of the vivarium are rarely fed; a few wood lice, centipedes and worms are added from time to time.

For class purposes, the vivarium has proved very useful, both for observation and for the storage of animals until they are wanted inside the laboratory. Thus collecting can be done all through the year, and it is not necessary to go out the night before a class has to be taken hoping to collect enough amphibious material.

For details of the cost, the reader is referred to the original article (*School Science Review* [59], March, 1934, p. 394).

## ZOOLOGICAL MATERIAL

### 33. COLLECTION AND CULTURE OF ZOOLOGICAL MATERIAL

*T. L. Green*

*Amæba*.—Examine samples of mud, bottom silt, etc., from slow-running ditches and ponds. They may sometimes be taken between two cover-glasses with a water film between them which are held together by a rubber band, dropped to the bottom of the water and buoyed by a cork at the surface connected by thread to the slips. Place garden soil in pond water and examine daily. Chop

up an earthworm in water and examine the water later. Chop up strands of the Canadian Water-Weed (*Elodea canadensis*) and cover with water, examine the surface scum in several days' time.

For culture, boil 20 wheat grains in 1 litre of water and cool the fluid; when cold, inoculate with *Amæba*. Add grains every month or so. Subculture once a term. It is wise to start off several cultures since they do not always "take."

*Euglena*.—Use net and also scrape mud and stones in stagnant green water of ditches and ponds. Common on estuarine mud flats.

For culture, add a little water containing *Euglena* to either of the following culture media, which are recommended by Dakin—

(a) Peptone . . . . .	0.5 gm.
Glucose . . . . .	0.5 "
Citric acid . . . . .	0.2 "
Magnesium sulphate . . . . .	0.02 "
Potassium phosphate ( $\text{KH}_2\text{PO}_4$ ) . . . . .	0.05 "
Water . . . . .	100 c.c.
(b) Ammonium sulphate . . . . .	0.2 gm.
Potassium phosphate ( $\text{KH}_2\text{PO}_4$ ) . . . . .	0.2 "
Magnesium sulphate . . . . .	0.1 "
Trace of iron sulphate ( $\text{FeSO}_4$ ) . . . . .	—
Tap water . . . . .	200 c.c.

*Paramecium*.—Chop up hay and simmer in water. Cool and then inoculate with *Paramecium*. Subcultures should be made from this about twice a term. It is said that cooling of the culture hastens conjugation, which should be looked for at frequent intervals.

*Volvox*.—As for *Euglena*, but swims free in water. *Volvox* often occurs in clear still water in well-lighted positions. May occur in temporary flood ponds formed on grass.

For culture, use methods for *Euglena*. Examine regularly for different stages of life history. Pond should also be examined throughout year.

*Vorticella*.—By careful examination of general aquatic plant debris and stems of small plants.

**Culture** As for *Euglena*. Also Agar's method—boil 3 gm. of fowl dung in 1 litre of water. Filter the fluid. Expose to sunlight. Use 1 part of above with 9 parts of filtered pond water. This solution supports a rich culture of green Protophyta on which *Vorticella* will feed. The *Vorticella* must be purchased or collected and added to the solution.

*General Instructions Relating to Protozoa.*—1 Living specimens can be concentrated for distribution to class by centrifuging, or making use of their reactions to light, thigmotaxis and other tropistic reactions. Material should be given out with a clean pipette and placed in covered watch-glasses. It is unwise to add tap water since the Protozoa are very susceptible to changes of pH.

2. Living material should always supplement permanent preparations if possible.

3. Observations of living Protozoa are facilitated by using dark ground illumination and vital stains.

4. Material should be observed in a cavity slide or the cover-glass should be supported by strands of cotton, etc.

5 In addition to complete observations of structure, much attention should be paid to functional observations of mode of movement, ciliary action and the contractile vacuole.

These observations may be aided as follows .

(a) Movement . . entangle the animal in strands of glass wool, pure wool, or by adding a little glycerine.

(b) Cilia . . . add dilute suspension of carmine or India ink.

(c) Vacuole . . run under the coverslip a drop of 0.25 per cent. Salt solution.

*Hydra.*—*Hydra* may generally be obtained by very carefully searching pond weed stems, plant debris from ponds containing an abundance of aquatic life, and is generally associated with duckweeds

*Hydra viridis* may be reared in a jar full of pond water containing minute organisms. *H. fusca* requires a good

supply of such food as *Daphnia* . . . place a few *Hydra* in a large jar of filtered pond water with a few strands of *Elodea canadensis*. Add *Daphnia* to this water at intervals. *Daphnia* may be easily cultured as follows :

(a) Add *Daphnia* to Agar's medium (see under *Vorticella*).

(b) To water containing *Daphnia* add a small amount of Horlick's Malted Milk Powder which forms a food pabulum.

(c) Use Taylor's method as for *Amœba*.

*Obelia* —The hydroid form may be found in rock pools attached by stolon to sea weed, rock surfaces, etc. Tow netting may provide the medusæ.

*Tænia* (Tapeworm).—Segments of a tapeworm can usually be obtained from dog fæces. Two forms are generally encountered. *Tænia serrata* and *Dipylidium caninum*. The scolex and a complete worm can usually only be obtained by post-mortem examination immediately after death. The "bladder-worm" of *T. serrata* is often encountered whilst dissecting the rabbit.

*Lumbricus* (Earthworm).—Worms may be taken by hand on grass and paths after dusk or after rain. They retreat quickly and care is essential to avoid breaking. They may be brought to the surface by "watering" with a thin mustard solution. They are best dug up with a garden fork.

A winter supply may be ensured by making a flat heap of leaf mould and light soil and placing captured worms therein ; cover this with leaves and sacking.

*Hirudo* (Leech).—*H. medicinalis* occurs comparatively rarely in ponds and streams in England. It should not be confused with the common "horse leech," nor with *Clepsine*.

Leeches should always be purchased alive. They will live in a clean jar of water for considerable periods without feeding. At intervals they may be presented with a piece of fresh raw meat.

*Astacus* (Crayfish).—Crayfish occur in many streams and

rivers. In clear water turn over stones and stand by with a net. It is said to be possible to take them on a line baited with a piece of raw meat.

Difficult to keep unless a good running water aquarium is available. By taking "berried" females the development may be followed. If hatching results, the young should be kept by themselves in clean running water and well supplied with aquatic creatures as food.

*Periplaneta* (Cockroach).—The cockroach is common in bakehouses, etc., and may be caught by hand or in a simple box trap.

Supplies may be kept alive in boxes with good ventilation. Provide the insects with rolls of paper for shelter and a constant water supply. Feed on bacon rind, fat, butter, bread, etc. Note egg cases: hatching does not take place until several months after laying.

*Asterias* (Starfish).—It is often possible to collect specimens from rock pools at the sea-side. They can also be obtained cheaply from fishermen since they often enter the lobster pots.

*Helix* (Snail).—*H. pomatia* has a wide but localized distribution in England. The common garden snail, *H. aspersa*, may be used, but is somewhat small.

If snails are kept warm and moist and well fed with fresh cabbage leaves, they do not hibernate.

## CHEMISTRY

### GENERAL MAINTENANCE

#### 34. METHOD OF FOLDING A FILTER PAPER

*E. J. Leeming*

A method of folding a filter paper so that a funnel may be dispensed with has been recently described (*Fuchs, Chem. Fabr.*, 1934, 7, 97, A, 1934, 750). The paper is first folded in half as in the usual method. It is then folded

along the lines AB and AC (Fig. 22) into three equal parts, and the two outer portions are bent over on opposite sides of the centre piece. It is next folded down the centre line AD (Fig. 23), first over on one side and then after re-

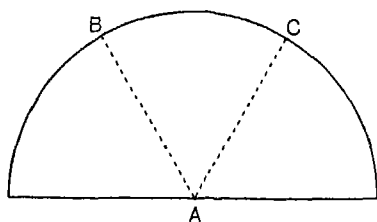


FIG. 22.

opening along the same line in the opposite direction. If it is opened to the state shown in Fig. 23 and a finger is inserted into the central division, i. e. between the two sides of BAC (Fig. 22), it will open into a small cone. On all sides the paper is in three thicknesses, which makes the

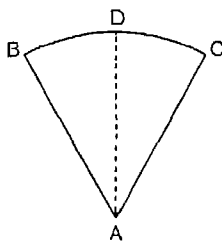


FIG. 23.

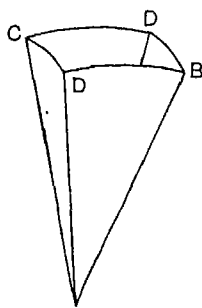


FIG. 24.

cone strong enough to hold liquid without a glass funnel to support it. Thus the paper may be placed in the top of a test-tube and small quantities of liquid may be filtered directly. This method might be found useful in qualitative analysis.

## 35. DEFLAGRATING SPOON

*K. H. Hagopian*

Special features <sup>1</sup>:

- (1) Heavy gauge of all parts.
- (2) The "boss" on the brass disc contains no cork—the  $\frac{5}{32}$ -in. iron rod just passes through a hole drilled in the "boss."
- (3) The disc can be fixed at any point along the iron rod by means of the side screw. (This screw has been made from a stainless iron rivet)
- (4) The cup has been "turned" in the lathe.

## 36. NICKEL BOATS

*K. H. Hagopian*

True one end of a bar of mild steel,  $1\frac{1}{2}$  in.  $\times$   $1\frac{1}{2}$  in. Mark off a length of  $2\frac{7}{8}$  in., using a steel square and scriber. Saw off, using a (hand) hacksaw. True up the end, using file and steel square.

Find the external diameter of the cross-section of the porcelain-boat and also its length. Use a twist drill of the same size ( $\frac{3}{8}$ -in.) and drill a hole lengthwise through the axis of the block of steel of the required length. There should be about  $\frac{1}{8}$ -in. of steel between bottom of hole and end of block. (Use a Power Drill (Electric Motor) if one is available.) Score the block lengthwise into halves by a scriber. Saw along scores, using hacksaw. True up the new faces.

(A) Take one half block; cut a piece of mild steel from a bar  $\frac{1}{4}$ -in.  $\times$  1 in (or  $\frac{1}{4}$ -in.  $\times$   $\frac{3}{4}$ -in.) to close open end of the groove. Screw it into place with  $\frac{5}{32}$ -in.  $\times$   $\frac{1}{2}$ -in round head Whitworth screws. Make guides from strip (or sheet steel)  $\frac{1}{16}$ -in thick. Screw them into position

<sup>1</sup> Made in the Engineering Laboratories, Edinburgh Academy. Shown at the S.M.A. Exhibition, Jan., 1934.

$\frac{5}{8}$ -in. apart with their inner edges equidistant from the edges of the groove

Cut a piece of  $\frac{3}{8}$ -in. mild steel rod to fit the groove, shaping the round end on an emery wheel or in the lathe. This bar should fit the groove loosely.

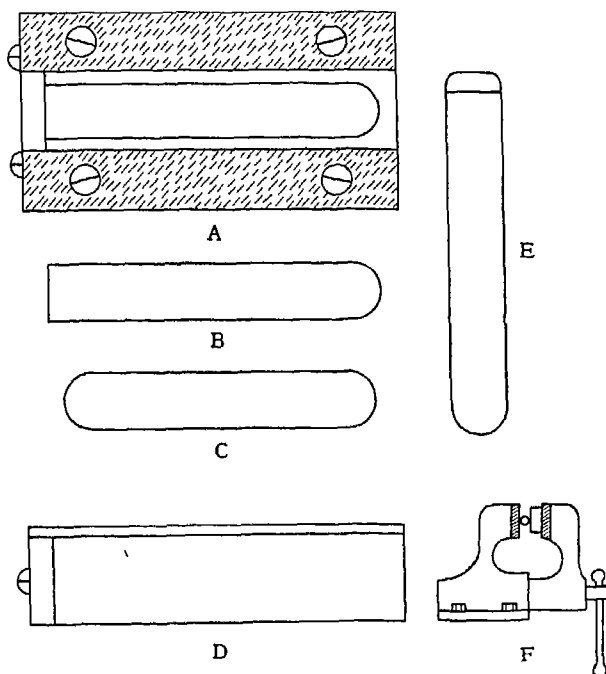


FIG. 25.

- A. Plan of mild steel block.
- B. Bar of mild steel for nickel boats.
- C. Bar of mild steel for stainless iron boats.
- D. Elevation of mild steel block.
- E. Nickel boat.
- F. Apparatus with nickel sheet in position ready for pressing.

Repeat from (A) for the other half block and thus make two formers.

Cut a strip of No. 28 gauge pure nickel sheet to fit between the guides on a block. Place the bar in position



on the top of the nickel. Set the whole in a parallel jawed bench vice and screw up firmly. Remove the "boat." Trim the round end and "tail" of the boat with snips; file smooth and then flatten "tail" with pliers. Stainless iron sheet may be used instead of nickel sheet. Then both ends of the bar should be rounded.

The cost of one nickel boat is less than  $1\frac{1}{2}d$ .

These boats are used for reduction experiments. When lead oxides have been reduced, most of the lead is removed by heating the boat red-hot, and shaking out. The rest of the lead is removed by further heating and shaking, followed by scraping, preferably with a suitably-shaped scraper, though a penknife may be used.

The advantages of these boats over the usual porcelain ones are: (1), cheapness; (2), they are easily cleaned; (3), they can be used many times without breaking.

### 37. REMOVAL OF RUBBER ADHERING TO GLASSWARE

*E. J. Leeming*

When rubber tubing has hardened and adhered to glass tubing, it is said (L Skazin, *Canad. J. Research*, 1934, **10**, 592. A, 1934, 861) that steam passed through the tube will soften the rubber and enable it to be removed easily.

### 38. SEALING WIRE INTO PYREX GLASS

*S. H. Piper*

Pieces of tungsten wire, short for economy's sake, are welded or brazed to another wire which serves as a handle. It is an advantage to use a vacuum melted wire for this purpose, and it is desirable to keep all wires that are to be used for leads as stout as possible.

The tungsten wire is cleaned in the flame by melting

sodium nitrite on it (this process is found to be more efficient than dipping in molten sodium nitrite as is usually recommended) and then oxidized in the flame to a blue colour. A loosely-fitting sleeve of pyrex tube is pushed over the tungsten and melted to it. Heating is to be continued until the tungsten inside the glass shows a golden-brown colour, due to the formation of a tungsten-boron compound on the surface of the wire. The colours attained in the various stages are extremely important—seals which do not show correct colours are very liable to crack—and can best be judged by an inspection of seals in the various stages of manufacture. The glass-coated wire can be joined on to an apparatus by the usual glass to glass technique, and any number may be placed in a row to form a pinch.

### 39. SEALING GLASS TO COPPER

*S. H. Piper*

Pyrex can be sealed directly on to copper tubes. The copper tube is turned down to a feather, about 1/1,000 in. thick, the upper edge of the feather is coated inside and outside with pyrex glass, and the seal made glass to glass in the ordinary way. The pyrex coating on the copper is strong enough to restrain the expansion of the thin copper feather. Such joints will stand repeated heating and cooling, can be used in liquid air and made in almost any size. Joints of more than  $\frac{1}{2}$ -in. diameter must be made on a glass-blower's lathe.

### 40. "SOLDERING" PASTE AND GENERAL CEMENT

*H. E. Watson*

Make collodion varnish by dissolving celluloid in amyl acetate and stir in aluminium powder.

The mixture sets quickly to a fairly hard mass, and since it is plastic in the intermediate stage, it can be moulded and worked neatly. It retains its pleasing metallic lustre permanently and has a neater appearance than ordinary soldering. The mixture has been used successfully for stopping a leak in a car radiator, and as a general adhesive.

#### 41. PROTECTION OF PHOTOGRAPHS AND DIAGRAMS

*R. Coles*

Photographs, illustrations, etc., which are likely to receive much handling in class may be preserved by placing them between a piece of card and a sheet of "Cellophane," turning over the edges of the cellophane and sticking them with seccotine.

The cellophane is moisture-proof and grease-proof; also the photograph may easily be removed if necessary since it is not stuck to the card. It is easier to use than glass. The cellophane is produced by The Cellophane Co., Ltd., 7, 8 and 9 Bird Street, London, W C.1, and sold by several stationers. Sheets, about 18 in.  $\times$  40 in., are about 3*d.* each.

#### 42. WATER BLOWER

*A. J. Mee*

The diagram (Fig. 26.) shows a simple water blower which will provide a sufficient blast of air to work a blow-pipe.

A wide glass tube, about 1 ft. long and 2 in. in diameter, is provided with a cork at one end through which pass the water outlet of a filter pump and an exit tube through which the air is driven. This tube is placed inside a

glass cylinder, such as a measuring cylinder. When the water is turned on, it partly fills the wide tube and overflows the outer cylinder. The whole apparatus is kept in the sink. The air blast, which is under a pressure of water equal to the difference in heights of the water inside and outside the wide tube, is regular, and can be used quite easily for working a blow-pipe instead of the ordinary foot-bellows. It can also be used to blow air through liquids, etc. The maximum pressure of the air-blast can be varied up to a limit, by increasing the length of the inner tube.

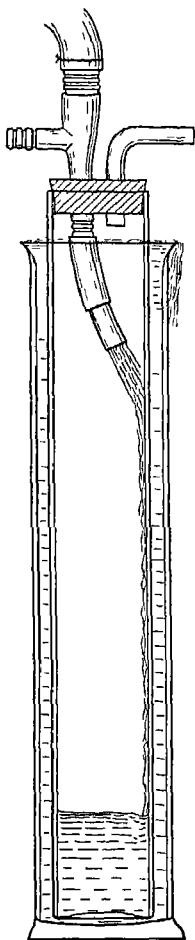


FIG. 26.

#### 43. FREEZING MIXTURES WITHOUT THE USE OF ICE OR SNOW

*W. Bryan Chivers*

During the summer months, a need often arises for a freezing mixture and no ice or snow is available. The following are well-tried recipes which are compounded of materials available in any laboratory. The lowest temperature reached is given, starting with ingredients and water at 12° C.

(a) Ammonium chloride . . . . .	5 parts	} — 12° C.
Potassium nitrate . . . . .	5 "	
Water . . . . .	16 "	
(b) Ammonium chloride . . . . .	5 parts	} — 16° C.
Potassium nitrate . . . . .	5 "	
Sodium sulphate cryst . . . . .	8 "	
Water . . . . .	16 "	} — 22° C.
(c) Ammonium nitrate . . . . .	1 part	
Sodium carbonate cryst . . . . .	1 "	
Water . . . . .	1 "	} — 40° C.
(d) Sodium sulphate cryst . . . . .	6 parts	
Ammonium nitrate . . . . .	5 "	
Nitric Acid (dilute) . . . . .	4 "	

#### 44. DEVICE FOR MAINTAINING THE GAS PRESSURE CONSTANT AT THE BURNER OF A GAS-HEATED THERMOSTAT

*F. Briers*

The arrangement is quite simple, consisting of a medium-sized jar *A*, through the tightly-fitting stopper of which passes a T-piece *B*, a thistle funnel *C*, and a second T-piece *D* shaped as shown. A small length of silica tubing connects with *D* by means of a piece of

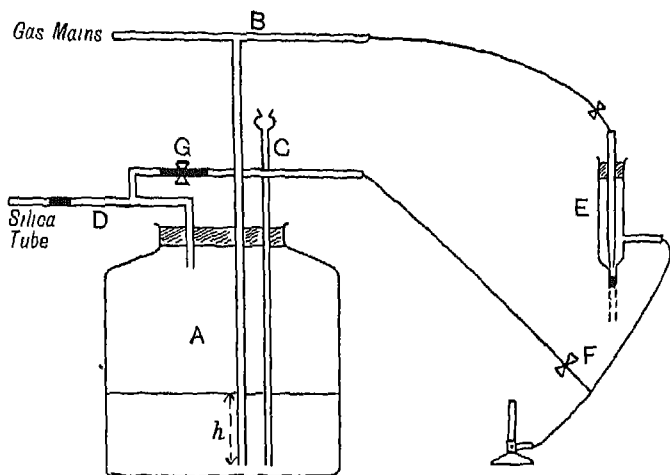


FIG 27

rubber pressure tubing, thus enabling gas constantly to be burnt. The other open end of *D* is similarly attached to a short length of glass tubing, the rubber pressure tubing in this case carrying an adjustable screw clip *G*.

Obviously, if *B* is connected to the gas main, the control tap being fully open, the height *h* in *A* measures the pressure at which gas is delivered to the burner. A very convenient value of *h* at which to work is 1–3 in. of water, since the normal main pressure is usually considerably greater than this maximum. Excess gas

simply bubbles through the water and is burnt at the silica jet. The height in A can be kept constant by addition of water through C.

During the heating period gas flows to the burner through the regulator E, the clips at F and G being adjusted according to the temperature at which the thermostat is running. During the cooling period gas cannot reach the burner *via* E, but sufficient can pass through G and F to keep it alight.

#### 45. USE OF MILK BOTTLES AS GAS JARS<sup>1</sup>

We have for the past twelve months used ordinary milk bottles as gas jars, without appreciating the difference. The tops can easily be ground on an emery wheel. The saving is considerable, the bottles costing 4s and the gas jars 12s a dozen.

#### 46. STORAGE OF GASES

*E. T. Harris*

By the aid of these vessels, which can be made from Winchester bottles, any gas that is not too soluble in water (such as oxygen, hydrogen, nitrogen, nitric oxide, carbon monoxide) can be kept for practically any length of time, and delivered when required into any ordinary apparatus. Since the gas stored is completely water-sealed, it cannot diffuse away however long it is kept. From one "Winchester quart" bottle one can obtain, e.g. nitric oxide

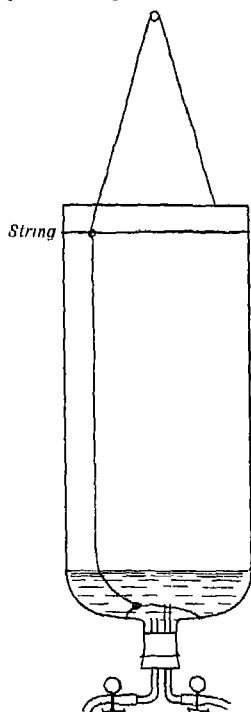


FIG. 28.

<sup>1</sup> Willesden County School Science Department per R. Hitchcock

sufficient to fill from four to six gas jars, and thus demonstrate its possible use for distinguishing nitrous oxide from oxygen, and its use in Priestley's eudiometer; or the oxygen from such a bottle can be used for the "Contact" process. When in use for storing gas, the bottles can be kept hung on the wall out of the way.

The Winchester is fitted up and strung as in the diagram, so that it may be used either way up. It is placed on its base, filled entirely with water through the longer tube, and the clips closed. It is then hung inverted, connected to the gas-making apparatus, and both clips opened. No pressure is required, so apparatus involving a thistle funnel may be used. The gas displaces the water, the clips are closed, and in this condition the gas may be kept (as in diagram). When the gas is required, the bottle is placed upright and water run in through the longer tube to force the gas out

## 47. GAS VOLUME MEASUREMENT

*H C Palmer*

The Ostwald gas burette consists of a cylindrical tube of 250-c.c. volume, graduated in cubic centimetres. It is mounted on a wooden upright and has a firm base. It is admirable for almost all the usual gas evolution experiments.

The burette is mounted on its board with thin brass strips and screws. Experience has shown that carefully made apparatus of this kind has a very long useful life.

One practical hint: Fill the apparatus until the water level is at zero graduation. Usually then, on attaching the gas evolution apparatus, the level in A falls. Before mixing the reacting substances, "pinch" the rubber junction tube away from the glass inlet tube and so, by creating a leak, restore the pressure in the apparatus to that of the atmosphere.

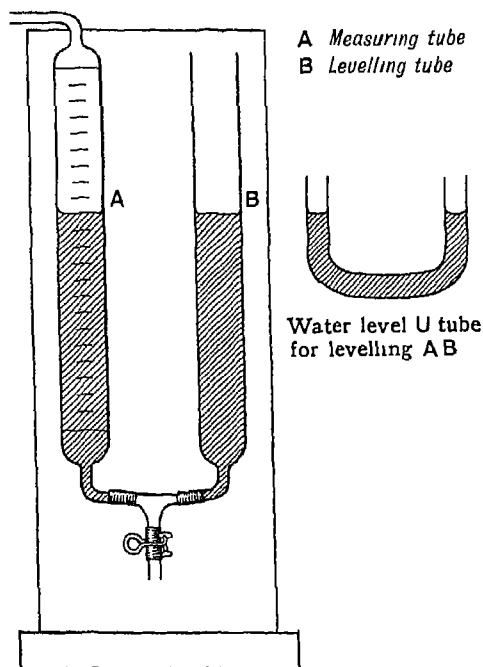


FIG. 29.

## 48. MODEL WATER SOFTENER

*E. J. Williams*

The model is constructed from a piece of glass tubing, approximately 40 cm. long and 4.5 cm. wide. A constriction is made about 6 cm. from one end, and a perforated porcelain plate is inserted. A similar constriction is then made at the same distance from the other end to support another plate. One-holed rubber bungs, tubes and a pinchcock are fitted as illustrated. The tube is packed between the constrictions, first, with a layer of gravel, about 3 cm. deep, then, nearly to the upper constriction, with permutite. The upper plate is inserted and covered with a layer of gravel, which serves to



distribute the water and to imitate large-scale filtration. The tap water is siphoned through the softener. The

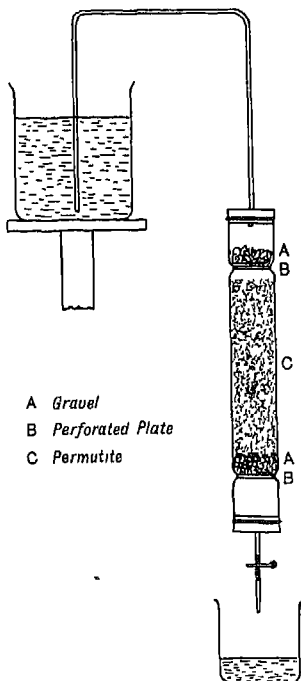


FIG 30.

permutite is regenerated by running 10 per cent. salt solution through, and the salt is washed away by distilled water. The following class results illustrate the efficiency of the softener :

50 c.c. of Newcastle-upon-Tyne tap water required 6.7 c.c. of soap solution.

50 c.c. of the same after boiling required 5.6 c.c. of soap solution.

50 c.c. after passing through softener required 0.6 c.c. of soap solution.

50 c.c. of distilled water required 0.5 c.c. of soap solution.

Without regeneration the apparatus will soften enough water for a form of twenty-four boys to perform sufficient titrations against soap solution to obtain agreement. The siphon-

ing should be commenced about half an hour before water is required.

#### 49. COLLECTING SPILLED MERCURY

*E. J. Leeming*

When mercury is spilled on a smooth surface, it is difficult to sweep it together because the droplets run about so easily. Professor C. V. Boys, in a letter to *Nature* (1934, 134, 29), recommends sprinkling the surface with water from a wash bottle. The drops do not

then run so easily and may be collected together with a piece of wood or cardboard.

## 50. PURIFICATION OF MERCURY

*F. Briers*

A large porcelain basin, which can be heated by a small Bunsen flame, acts as a reservoir for the mercury to be cleaned. By connecting E to a good water pump, and by regulating the clip on the piece of rubber A, an air-lift for the mercury to the bulb C is secured. B is a piece of stout rubber tubing connecting the air-lift to C. C acts as a splash-head, the air taken in passing *via* E to the water pump, whilst the mercury drops into the fall-tube D and is returned to the reservoir. This simple apparatus, in addition to securing an automatic circulation of the mercury, provides an excellent method of oxidizing the impurities usually present in ordinary laboratory samples of mercury.

After running the apparatus as long as necessary for the purpose in hand, the scum is filtered off by means of chamois leather. The length of the fall-tube D obviously needs to be 80-90 cm.

This device for raising mercury to a height greater than 76 cm. can readily be adapted to a Sprengel pump, thus ensuring its automatic action.

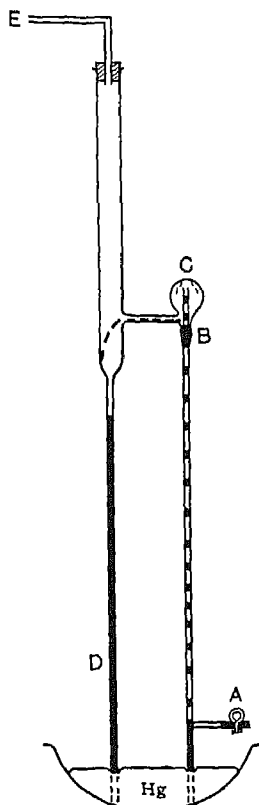


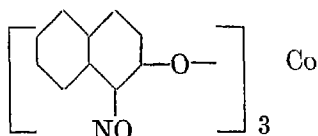
FIG 31

## ANALYSIS—QUALITATIVE

## 51. SPECIFIC PRECIPITANTS

*W. Cule Davies*

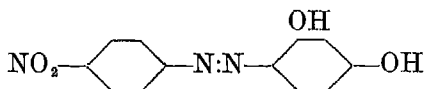
*Cobalt.*—(a) One of the earliest specific precipitants used was  $\alpha$ -nitroso- $\beta$ -naphthol (Ilinsky and von Knorre, 1885). When a solution of the precipitant in 50 per cent. acetic acid is added to a solution of a cobaltous salt acidified with hydrochloric acid and heated to  $80^\circ$ , a purple-red precipitate of the constitution



is thrown down. Under these conditions nickel is not precipitated.

(b) A hot, aqueous solution of phenylthiohydantoic acid,  $\text{NH}_2\text{C}(\text{N}.\text{C}_6\text{H}_5).\text{S}.\text{CH}_2\text{COOH}$  (Willard and Hall, 1922), is added to a hot solution containing cobalt and a little free ammonia, when a precipitate of the cobalt salt is obtained. The reagent forms a means of separating cobalt from nickel, iron and metals other than those of the hydrogen sulphide group. With iron present, ammonium citrate is added

*Magnesium*—A blue colour or precipitate is obtained when a solution of resorcin azo-p-nitrobenzene,



(Suitsu and Okuma, 1926), in dilute sodium hydroxide solution is added to a solution containing magnesium. Nickel and cobalt should be first removed by precipitation as sulphides.

*Sodium.*—The reagent (Kahane, 1930) consists of magnesium uranyl acetate solution made as follows. Heat

on a water-bath 32 gm. of uranium acetate, 100 gm. of magnesium acetate, 20 c.c. of acetic acid, 500 c.c. of 90 per cent. alcohol, and 300 c.c. of water. Cool, make up to 1,000 c.c. with water, allow to stand and filter. Preserve in coloured bottles. When the reagent is added to the cold, neutral, weakly acid or alkaline solution containing a sodium salt, a crystalline precipitate,  $\text{NaMg}_3(\text{UO}_2)(\text{CH}_3\text{COO})_9 \cdot 8\text{H}_2\text{O}$ , is obtained. With the exception of the orthophosphoric ion, other anions and cations do not interfere.

*Nitrate.*—When a nitrate solution is carefully added to either diphenylamine or diphenylbenzidine in sulphuric acid solution, a blue ring appears at the junction of the liquids. These tests are of extreme sensitivity.

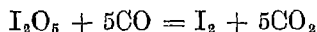
### ANALYSIS—QUANTITATIVE

#### 52. ESTIMATION OF SMALL QUANTITIES OF CARBON MONOXIDE IN AIR

*E. J. Williams*<sup>1</sup>

The estimation of fairly large proportions of carbon monoxide by the use of solutions of cuprous chloride is well known, but, whereas most text-books state that small quantities of the gas may be estimated by its reaction with iodine pentoxide, the method to be employed is not usually given, and in some cases such details as are supplied are misleading.

The reaction is



and takes place at *c.* 160° C.

The air in which carbon monoxide is to be determined is collected in the gas-holder A, and the method of

<sup>1</sup> For the experimental details, the writer is indebted to F. P. Mills, Esq., Director of the Northumberland and Durham Collieries Rescue Station. These details have been varied in the methods of measuring the air, drying the gas, and the heating of the iodine pentoxide.

measurement employed was to run a known volume of water from the upper reservoir into the gas-holder. Obviously the gas-holder could be replaced by an aspirator made from a Winchester bottle. The air must be bubbled through very slowly. The Dreschel bottles B and C contain respectively fuming sulphuric acid for the absorption of ethylene, and caustic potash solution for the removal of acid. The gas must be thoroughly dried, and this is achieved by caustic potash sticks and calcium chloride in the tower D, followed by phosphorus pentoxide in the U-tube E. To prevent choking, the phosphorus pentoxide is interspersed with glass wool.

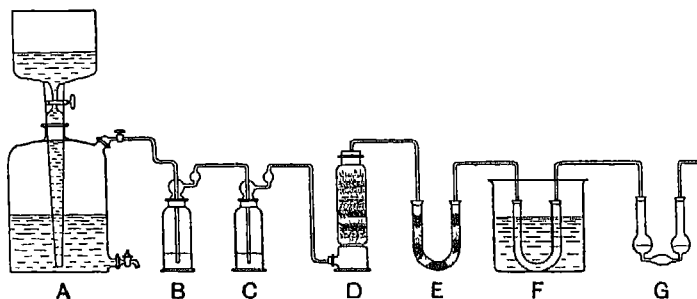


FIG. 32

Sufficient iodine pentoxide (B.D.H.) to reach about half an inch above each side of the bend is contained in the small U-tube F, which is heated to *c.*  $160^{\circ}\text{C}$  in a beaker containing medicinal paraffin. A method of heating which has proved successful has been by means of an electric immersion heater (300 watts) and the proper temperature was maintained by operation of a switch as required. Constant watching and stirring are, of course, necessary.

The bulb U-tube, G, contains potassium iodide solution for the absorption of the liberated iodine. Most of this, however, condenses in the connecting tube between F and G. It is transferred to G by dipping the end of the connecting tube nearer to F into a beaker

containing potassium iodide solution and sucking through the other end of G.

The iodine is then titrated against *N*/100 sodium thiosulphate solution.

1 c.c. *N*/100  $\text{Na}_2\text{S}_2\text{O}_3 \equiv 0.56$  c.c.  $\text{CO}$ .

### 53. ESTIMATION OF ENOL FORM IN ACETO-ACETIC ESTER

*L. G. Defoe*

Prepare the following solutions :

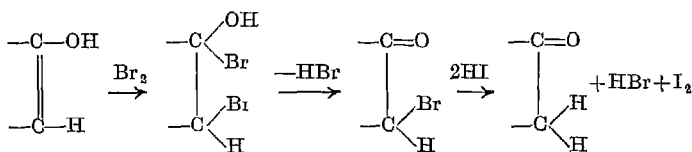
(1) 3 gm acetoacetic ester dissolved in alcohol and cooled in an ice-salt freezing mixture.

(2) 0.2 c.c. bromine in 10–20 c.c. alcohol, cooled in ice-salt freezing mixture

(3) 1.6 gm.  $\beta$ -naphthol in 20 c.c. alcohol.

(4) 1.3 gm. KI in water.

Add (2) and (3) in rapid succession to (1), followed by (4). The enol form is brominated (excess bromine being taken up by the  $\beta$ -naphthol), loses a molecule of  $\text{HBr}$ , and then liberates a *molecule* of iodine. The iodine is estimated by titration against  $\frac{N}{10}$  thiosulphate.



whence 254 gm.  $\text{I}_2 \equiv 130$  gm enol form.

The percentage of enol form at room temperature is about 3.

(*N B.*—Owing to the high temperature coefficient of tautomeric change,  $-10^\circ \text{C}$ . is sufficient to freeze the equilibrium, and the formation of more enolic form to replace that converted to keto is prevented.)

(The above method is due to Kurt Meyer, and the

quantities were obtained from the Balliol Laboratory, Oxford.)

### ANALYSIS—VOLUMETRIC

#### 54. USE OF ZINC AMALGAMS FOR REDUCTION WORK IN VOLUMETRIC ANALYSIS

*R. Groves*

Liquid amalgams as reducing agents were introduced by Nakazono (*J. Chem. Soc. Japan*, 1921, **42**, 526, 761), and zinc amalgam, in particular, has been in use in many advanced laboratories for some time. The amalgam is of particular use in the estimation of iron as in a mixture of ferrous and ferric sulphates, or of a mixture of cupric, ferric, and ferrous sulphates; these mixtures are commonly set as problems. A decinormal solution of a ferric salt can be reduced in this way in less than one minute, and since the solution can be decanted from the amalgam, the difficulty of filtering made necessary by the use of, say, zinc dust, with possible oxidation, is avoided. Copper sulphate is reduced in approximately one minute to metallic copper which conveniently dissolves in the amalgam, facilitating the estimation of the second mixture mentioned.

A suitable zinc amalgam is very conveniently made, simply by covering 200 gm. of mercury with a little dilute sulphuric acid and adding about 5 gm. of zinc. It is kept in a small reagent bottle with a good stopper, under very dilute sulphuric acid. The zinc can be renewed when the amalgam becomes diluted after considerable use. Zinc amalgam will bring about reduction of.

ferric to ferrous iron,  
permanganate to manganous salt,  
dichromate or chromate to chromium salt,  
cupric salt to copper metal (which dissolves in the amalgam).

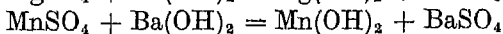
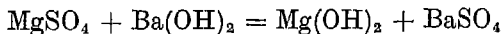
Of the less common reagents, it reduces penta-valent to di-valent vanadium, enabling the successive colours of the quadri- and ter-valent states to be seen. Titanic sulphate is completely reduced to titanous, this being a very convenient method of preparing titanous sulphate solution for volumetric analysis. The sulphates of the metals are generally used.

The best method of procedure is to take an aliquot portion of the solution to be reduced, and transfer it to the stoppered bottle containing the amalgam. The whole is then vigorously shaken by hand for one minute, and the reduced solution transferred to the titration flask. The amalgam is shaken up with two successive portions of about 20 c.c. of dilute sulphuric acid, and these are decanted and added to the reduced solution, care being taken in each case to see that no amalgam is poured over with the solution. As was shown by Russell (*J.C.S.*, 1926, 497) reduced solutions of this kind—ferrous, titanous, etc.—are perfectly stable in air at ordinary temperatures if the acid strength is of the order of twice normal.

## 55. METHODS OF VOLUMETRIC ANALYSIS EMPLOYING ADSORPTION INDICATORS

*A. W. Wellings*

*Sulphates.*—Normal and decinormal solutions of magnesium and manganese sulphates may be titrated directly with N/5 barium hydroxide solution using 1 per cent. alcoholic fluorescein as an adsorption indicator. In the reactions :



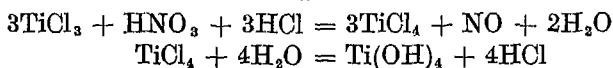
two precipitates are produced, and the colour change, yellow  $\rightarrow$  pink  $\rightarrow$  orange (on shaking), is due to adsorption of ions on the surface of the hydroxide and not on



the barium sulphate. Consequently the method can be extended to other sulphates and alums by providing manganous or magnesium hydroxide in the titration. In estimating other sulphates, 5 c.c. of 1 per cent manganous (or magnesium) acetate and 5 drops of the indicator are added to every 25 c.c. of sulphate solution and the mixture titrated with N/5 barium hydroxide. Excellent results are obtained with all sulphates except those which form deeply-coloured hydroxides, though, curiously enough, no difficulty is experienced with nickel sulphate: mercuric sulphate cannot be estimated, although it gives a vivid colour change, owing to the formation of yellow basic mercuric sulphate during the titration. The presence of H ions causes inaccurate results, since the first additions of barium hydroxide are employed in neutralizing the acid: consequently those sulphates which are acid in solution owing to hydrolysis must be neutralized before titration with barium hydroxide. On the other hand, the presence of OH ions does not affect the accuracy of the determination.

*Oxalates.*—Decinormal and centinormal solutions of normal oxalates may be titrated directly, at room temperature, with a standard solution of lead acetate using fluorescein as an adsorption indicator. The oxalate solutions must be neutral, thus, the method is unsuitable for the titration of binoxalates and potassium quadroxalate: the end-point is denoted by the permanence of the pink coloration on the particles of lead oxalate formed in the titration.

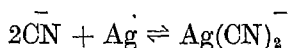
*Nitrates.*—Nitrates may be estimated rapidly and accurately in decinormal and centinormal solutions by titration with standard titanous chloride. Alizarin is used as the adsorption indicator, the colour change occurring on the particles of titanous hydroxide formed by hydrolysis, on boiling, during the titration. The general reaction may be represented by the equations:



The standard solution of titanous chloride is prepared in exactly the same way as for a ferric salt determination, and the standardization and the subsequent titrations must be carried out in an atmosphere of hydrogen in the usual manner. The method of nitrate titration is as follows: 25 c.c. of decinormal nitrate solution are diluted about three times with water (to assist the hydrolysis of  $\text{TiCl}_4$ ) and 5 drops of 1 per cent. alcoholic alizarin added: the mixture is then boiled and titrated with the standard  $\text{TiCl}_3$  solution. The addition of the first few drops of  $\text{TiCl}_3$  produces a deep greyish-green colloidal solution, the colour of which disappears on boiling, the original red colour being restored: the solution must be boiled after each addition of  $\text{TiCl}_3$  and, as the titration proceeds, a precipitate is formed, on the particles of which the colour change occurs. The titration is continued until the original red colour is no longer restored on boiling, the end-point being denoted by the permanent grey colour of the mixture. All nitrates, with the exception of those nitrates which form basic salts and uranyl nitrate, may be estimated in this manner: uranyl nitrate forms no precipitate in the titration, probably owing to the formation of titanium uranate or diuranate. The estimation may also be carried out with centinormal solutions of nitrates provided that adequate volumes are titrated: for example, 50 c.c. of a centinormal nitrate solution will be equivalent to 15 c.c. of 0.1 molar  $\text{TiCl}_3$ , it is inadvisable to employ solutions of  $\text{TiCl}_3$  in concentrations weaker than 0.1 molar, since a good yield of titanic hydroxide is essential in the titration. If sulphuric acid be used, instead of hydrochloric acid, in the preparation of the standard solution of  $\text{TiCl}_3$  negative results are obtained: this may be due to the fact that titanium forms a basic sulphate. The solutions of  $\text{TiCl}_3$  used by the author were prepared as follows: 200 c.c. of commercial 15 per cent.  $\text{TiCl}_3$  solution were mixed with 400 c.c. of concentrated  $\text{HCl}$ , and diluted to 1 litre; on

standardization this yielded a solution about 0.14 molar.

*Alkali Cyanides.*—Diphenyl carbazide may be used as an adsorption indicator in Liebig's original method for the determination of alkali cyanides by direct titration with standard silver nitrate. The colour change, pink  $\rightarrow$  violet, occurs on the particles of silver cyanide, precipitation of which begins as soon as silver ions are present in excess of the concentrations required by the equation



Very accurate results are possible in centinormal solution: in fact, millinormal solutions of cyanide may be used, but it is inadvisable to use solutions of silver nitrate more dilute than N/250. The silver nitrate solution must be neutral

*Lead Salts.*—Lead nitrate and lead acetate may be determined very rapidly in M/10 and M/100 concentrations by titrating standard NaOH with the lead solution. Fluorescein, dichlorofluorescein, and dibromofluorescein may be used as adsorption indicators. The end-point is indicated by the permanence of the pink colour on the precipitate of lead hydroxide. Five drops of 0.1 per cent. alcoholic solution of the indicator are sufficient in titrating 25 c.c. of the lead solution. It is important to note that the lead solution must be run into the standard NaOH, and not vice-versa. The method is excellent for the standardization of the lead acetate solution required in the titration of oxalates, and borates.

*Borates.*—Borax, sodium metaborate, and sodium "perborate" may be determined in aqueous solution by direct titration with standard lead acetate solution, using dichlorofluorescein as an adsorption indicator. The colour change, yellow  $\rightarrow$  pink, occurs on the particles of lead metaborate precipitated in the titration. Five drops of 0.1 per cent. alcoholic indicator solution are used in titrating 25 c.c. of borate solution. For very accurate results, the borate solutions must first be

neutralized with nitric acid, but reasonably accurate results are obtained without this precaution

## REFERENCES

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*Analyst*, 1933, 331.

## COMBUSTION AND OXIDATION

56 LAVOISIER'S EXPERIMENT ON THE  
CALCINATION OF MERCURY

*W. Wheatley*

The retort or matrass can be procured through the laboratory furnisher; the body of the retort is made from a round-bottom flask of about 600–700 c c. capacity, to which is attached a tapering neck, 14 to 16 in. long, of about  $\frac{1}{2}$ -in. diameter at the narrow end. The glass cover for an electric bulb serves as a suitable bell jar, dimensions approximately 6 in.  $\times$  3 in., and a glass pneumatic trough, 8 in.  $\times$  4 in., is of a convenient size. The jar is supported on a shelf made from a piece of sheet lead which can be cut and bent to the appropriate shape.

It is not essential that the volume of the air enclosed should be exactly 50 cu. in. (50 Old French cu. in. = 990 c c), though it is desirable in the interests of historical accuracy that it should approximate to this quantity

In order to allow for the gradual addition of water to the trough as the experiment proceeds, the initial level should be about  $1\frac{1}{4}$ – $1\frac{1}{2}$  in. below the rim. Provision must also be made for the expansion of the air in the early stages of the experiment; the 50-unit mark should be at least 1 in. from the base of the jar. The leaden shelf can easily be cut or bent to allow of this adjustment. Having fixed the position of the 50-unit

mark, find the total volume of air enclosed in the apparatus; the volume of air in the retort is best determined by finding the weight of water required to fill it. Now calculate in c.c. the value of 40 units; mark this position on the paper scale and divide the space between the 50 and 40 into four equal parts, numbering them 47.5, 45, and 42.5. Smaller divisions are undesirable since they will confuse the eye; cover the scale with wax and dry the retort thoroughly before commencing the experiment.

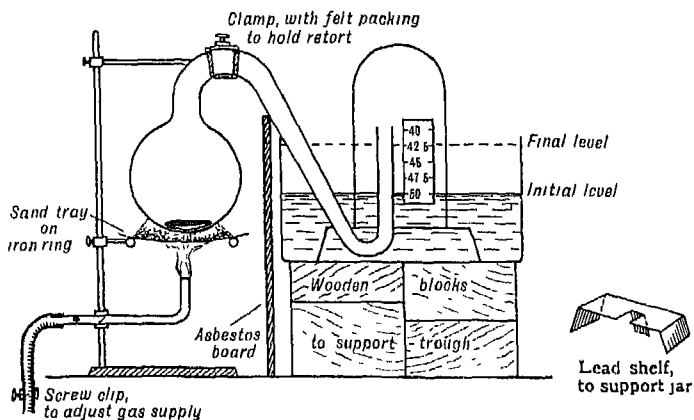


FIG. 33.

Pour into the retort 4 oz. mercury, approximately 122 gm. By means of a bent tube adjust the levels of the water so that when the experiment begins 50 units of air are enclosed. The retort may be heated over a sand-bath by means of a very small Bunsen flame. It is advisable to turn off the gas for about an hour every day so that the class may see how the volume of air diminishes; moreover, this allows for the diffusion of unused oxygen from the jar to the retort. The red oxide is visible at the end of the second day.

Lavoisier's directions for conducting the experiment are so explicit that further description is unnecessary,

but it is doubtful whether it is worth while collecting the oxide in order to measure the oxygen evolved. Much of the oxide adheres tenaciously to the inside of the retort and can only be removed by breaking the glass. If the retort is cleaned with a little dilute nitric acid, it can be used over and over again, and after the first occasion the experiment can be set going with the minimum of trouble.

The results agree closely with Lavoisier's figures. He said that after allowing for changes of temperature and pressure, 7-8 cu. in. of air were used up. The reaction is, of course, reversible; a state of balance seems to be reached after twelve days, for on further heating, there is no further diminution of volume.

#### 57. INCREASE OF WEIGHT ACCOMPANYING COMBUSTION OF A CANDLE

*E. J. Sumner*

A simple method of demonstrating that the products of combustion of a burning candle weigh more than the substances originally present can be conducted as follows. Invert over the lighted candle on a balance pan a wide-mouthed conical beaker (tall form), the bottom of which has been moistened with caustic soda solution and sprinkled with dry powdered slaked lime. This is very simple and answers very well.

#### 58. COMBUSTION OF HYDROGEN

*R. G. Reid*

Instead of allowing the burning jet of hydrogen to impinge on a flask through which cold water runs, a tube about 1 in. in diameter and about 2 ft. in length can be used as a condenser. Sufficient moisture will collect on the cooler parts of the tube to drop on to a watch-glass.

## 59. HYDROGEN-OXYGEN MIXTURE

*R. R. Finney*

Bubble electrolytic gas through a good soap solution. The soap bubbles float away, and a lighted taper brought near causes a loud but harmless explosion. This is a much better demonstration than the usual detonating flask experiment.

## 60. GLASS BUNSEN BURNER

*E. J. Harris*

A glass tube about 10 mm. diameter is attached to the gas supply by rubber tubing and the gas ignited.

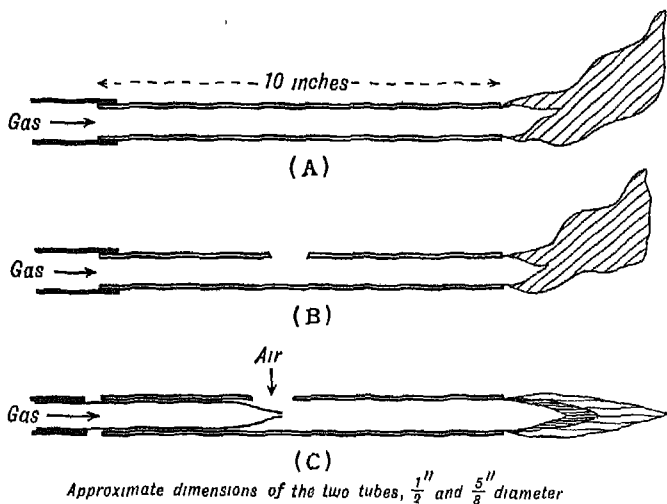


FIG. 34.

A smoky luminous flame is produced (A) A hole is now blown in the side of the glass tube, with the object of allowing air to enter the gas stream, so rendering the flame hotter and less luminous. The gas is again ignited, but the character of the flame is found to be unchanged

(B). However, on introducing a slightly-narrower glass tube about 8 mm diameter provided with a jet, the flame changes completely and assumes the familiar "Bunsen" form (C). By moving the narrower tube to and fro inside the wider tube, considerable variations in the character of the flame can be produced.

The experiment shows the importance of the jet in the construction of a Bunsen burner.

## 61. STRIKING-BACK OF A BUNSEN BURNER

*Allan Adair*

The striking-back of a Bunsen burner may be demonstrated to a large audience by the following apparatus, which is both simple to construct and spectacular to watch.

A is a glass tube, 100 cm. long by 2.5 cm. wide, clamped vertically above a Bunsen burner. A narrow glass tube, S, is attached to A by string. The upper end of S is bent as shown and drawn out into a jet, J. The lower end of S is attached to the gas supply and the jet is lighted. This flame is used to ignite the explosive mixture which ascends A.

It is important that the jet is arranged so that it lies about  $\frac{1}{2}$ -in. from the rim of the tube A, since after the explosion the spent gases tend to extinguish the igniting flame.

At G is arranged a piece of copper gauze which is pressed as firmly as possible across the tube and around its walls (gauze with 28 meshes to the inch will do). This gauze cage serves to prevent the flame travelling down to the burner, the top of which should be about 2 in. below the gauze.

With the air holes of the burner closed, and the gas

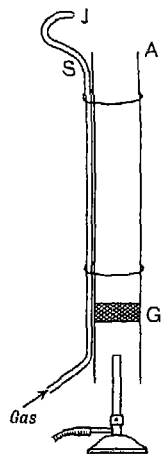


FIG. 35



turned fully on, a steady flame should ignite at the top of A. The air holes are now opened slowly until a large Bunsen flame, with a bright blue inner cone an inch high, is obtained. The gas is now slowly turned off until a strike-back takes place, the flame travelling down the tube until it is extinguished at G. A fresh supply of explosive mixture now ascends until it is ignited at J. Successive explosions take place. The apparatus runs very steadily when it has become warm after the passage of the first two or three flames, and may be run without attention for some minutes without becoming too hot. It is perfectly safe in operation.

With this apparatus the principle of the Fisher burner may also be demonstrated. If an ordinary gauze is held over the upper end of the tube, the flame is prevented from striking back, and burns quietly above the gauze which conducts and radiates the heat away from the flame, so that the temperature of the ascending mixture does not reach the ignition point below the gauze.

## 62 ILLUSTRATION OF THE PRINCIPLE OF THE DAVY LAMP

*F. Fairbrother*

A copper gauze cylinder, closed at one end, is made from a piece of gauze of about 30 mesh,  $6\frac{1}{2}$  in.  $\times$  6 in. This produces a cylinder  $5\frac{1}{2}$  in. high and  $1\frac{1}{2}$  in. diameter, with  $\frac{3}{4}$ -in. overlap. Along the top edge V-cuts are made,  $\frac{1}{2}$ -in. down. To close the top, a disc of gauze is inserted and the top edges folded over. The cylinder is bound with a few turns of bare copper wire, S.W.G. 22, or thereabouts.

The cylinder is pressed down over a piece of candle,  $1\frac{1}{2}$  in. long, in a circle of plasticene, 4 in. diameter and  $\frac{1}{2}$ -in. thick. The gas from a Bunsen burner can be played on the outside of the cylinder while the candle

is alight inside. The coal gas can be observed burning inside the gauze, but the burner is not ignited.

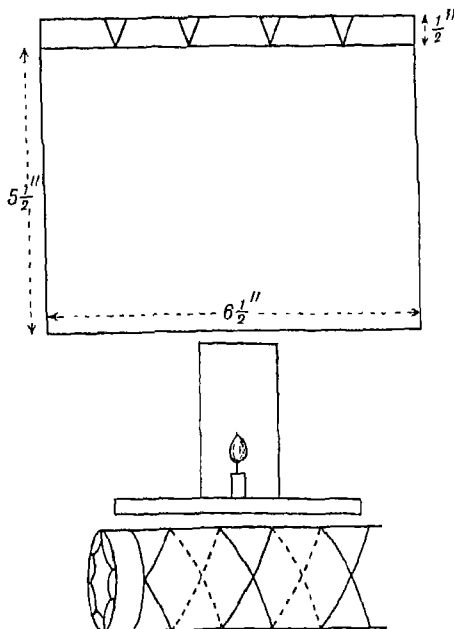


FIG 36.

Repeat, after making a small depression or tunnel in plasticene with a glass rod. the Bunsen burner ignites.

### 63. PYROPHORIC ZINC <sup>1</sup>

*S. A. Coase*

About 50 gm of zinc dust, of the quality used for laboratory reductions, are moistened with 10 per cent. caustic soda and thoroughly mixed to form a stiff paste. Excess moisture is then removed by pressing between filter papers. The mass is then placed on a sheet of asbestos board and exposed to the air of the laboratory. After

<sup>1</sup> Sebborn, *Trans. Faraday Society*, No. 144, Vol. XXIX, 1933.

about ten minutes, steam begins to rise rapidly, and after fifteen minutes, the mass commences to glow brightly with vigorous oxidation, the incandescence spreading rapidly.

The mechanism appears to be as follows. Sodium hydroxide dissolves the surface film of oxide and hydroxide from the metallic zinc, forming sodium zincate. The combined effects of the heat of this reaction and the removal of the protecting oxide film from the naturally reactive zinc causes further oxidation to take place: the resultant new oxide film is again removed by the caustic alkali; hence the reaction  $\text{Zn} + \text{O} \rightarrow \text{ZnO}$  proceeds with acceleration, until the temperature is sufficient to cause incandescence.

### COMPOSITION BY VOLUME

#### 64. PROPORTION OF OXYGEN IN AIR

*F. B. Field*

The following simple experiment, suggested by one of the boys during a lesson on oxygen to the Third Form, may be of interest.

Two gas jars of exactly equal capacity were filled, one with oxygen and the other with air. At a given signal, sulphur burning in a deflagrating spoon was lowered into each jar, and the time at which each flame was extinguished was noted. The jar of air supported combustion for twenty-one seconds; the burning continued in the oxygen for ninety-six seconds. The results of a rather rough-and-ready experiment are surprisingly near the five to one ratio.

#### 65. PROPORTION OF OXYGEN IN AIR BY PARTIAL PRESSURE

*F. Fairbrother*

Into a thin glass test-tube, 5 in.  $\times$   $\frac{5}{8}$ -in., introduce 2 pellets of pyrogalllic acid (use B.W. Tabloids or 0 13 — 0 2

gm.) and about  $\frac{1}{2}$ -in. of a stick of caustic potash. Pull out the test-tube as shown in Fig. 37 (A). Nearly fill the lower portion of the test-tube with water by means of a dropping tube. Withdraw the dropping tube and seal off the bulb A. Introduce the bulb (sealed end downwards) and its contents into the pyrex tube T (8 in.  $\times$  1 in.) which is full of air and saturated with water vapour (merely by having the tube wet inside); see Fig. 37 (B). Insert the rubber stopper, open the clip A and pour

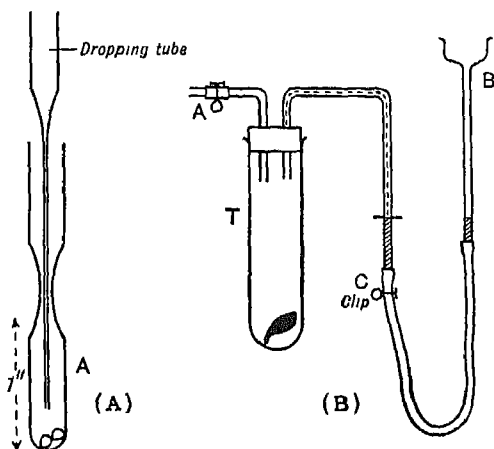


FIG. 37.

mercury into the manometer tube B. Note carefully the height above the level of the bench of the mercury in the tube B and put a strip of gummed paper at the level in left arm. Close clip A. Pinch the rubber tubing by the screw clip C (to prevent shaking the mercury from the manometer during the next part of the experiment) and give a fairly vigorous shake to test-tube T. This breaks the bulb and the solution of pyrogallie acid in caustic potash begins to absorb the oxygen. Shake the test-tube gently for three to four minutes so that all the oxygen is absorbed. Open the clip C. The mercury is seen to rise in the left arm of the manometer. The tube B is now lowered until the mercury in the left

arm is brought to its original level. The height of the mercury in B above the bench is again measured and results obtained, set out as follows :

Barometric pressure . . . . .	75.5 cm.
Height in B before absorption . . . . .	44.6 cm.
Height in B after absorption . . . . .	29.6 cm.
Diminution in pressure due to absorption of O . . . . .	15.0 cm.
This partial pressure = $\frac{15}{75.5} = \frac{1}{5}$ approx.	

## 66. COMPOSITION OF WATER BY VOLUME

*W. B. Barker*

The classic experiment in which 2 volumes of hydrogen and 1 volume of oxygen are exploded in a eudiometer surrounded by amyl alcohol vapour is one which presents certain difficulties under ordinary school conditions. Owing to the inflammable nature of the amyl alcohol vapour, its use is not always advisable. It has been the practice of the writer, after demonstrating the 2 : 1 ratio by explosion of hydrogen and oxygen at the ordinary temperature, to carry out the above experiment by using steam instead of amyl alcohol vapour.

With a mixture of 2 volumes of hydrogen and 1 volume of oxygen, the use of a steam jacket is inadvisable since there is a danger of the steam produced by the explosion condensing momentarily and of the eudiometer being broken by the rebound of the mercury. In the presence of even a small excess of hydrogen, however, there is little, if any, danger of this steam condensing even if the temperature of the jacket drops a little below 100° C. for its partial pressure is less than 1 atmosphere and, moreover, the excess hydrogen remains to act as a cushion.

The *modus operandi* is as follows :

Hydrogen is admitted to the closed limb until about  $1\frac{1}{2}$  in length of mercury is displaced. The temperature

of this gas is brought to  $100^{\circ}\text{C}$ . by passing steam through the jacket, and the pressure made equal to that of the atmosphere. The volume of the hydrogen is marked by a thin strip of gummed paper (A) placed level with the top of the mercury on the outside of the steam jacket.

A much smaller volume (about a couple of bubbles) of oxygen is then introduced. When the temperature is again at  $100^{\circ}\text{C}$ . and the atmospheric pressure has been restored, a second piece of gummed paper (B) is put on to mark the volume of oxygen added. A third piece of paper (C) is placed above the first piece at a distance twice that separating the two lower strips A and B (see diagram). It is then explained to the class that the volume of gas between the two upper strips (C and A) represents the 2 volumes of hydrogen which will combine

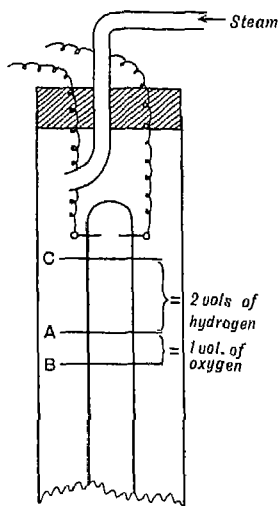


FIG 38

with the 1 volume of oxygen represented by the distance between the strips A and B; also that for the purpose of the experiment, the excess of hydrogen may be neglected. After explosion and restoration of atmospheric pressure, the mercury is found to have risen to the strip A and the conclusion is thus reached that the volume of the steam formed occupies the same volume as that of the 2 volumes of hydrogen which combined with the 1 volume of oxygen.

## 67. COMPOSITION OF OZONE

*G. H. Locket*

A very simple apparatus for finding the formula of ozone can be made as follows A piece of wide tubing

(internal diameter  $\frac{3}{4}$ -in.) is first drawn out carefully (A, Fig. 39) to make a narrow tube (about 2 mm. diameter), whose uniformity can be tested with a mercury pellet; or a piece of narrow tube can be sealed on to a piece of

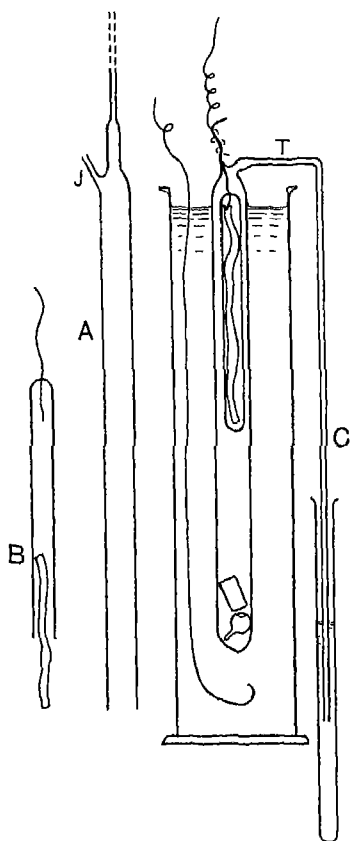


FIG. 39.

intermediate size and this attached to the big piece. The narrow tube will presently serve as a manometer. At a spot near the junction (J) the glass is melted, touched with another piece of glass and drawn out into a narrow neck, into which the wire for the inside electrode is to be sealed. This electrode is made by enclosing a piece of aluminium foil from a cigarette packet in a narrower tube, 8-9 cm. long (B), and making contact with a wire sealed in at one end, which is passed up into J and sealed in again.

A thin capsule of turpentine and a marble or piece of glass rod are now introduced, the bottom of the wide tube drawn out and the narrow tube bent round as shown (C). A stream of dry oxygen is passed through, and when

the tube is full, the bottom is sealed off and the apparatus left in a gas jar of melting ice (the outside electrode). When the liquid level in the manometer tube, which dips into concentrated sulphuric acid, is steady, the current from the coil is applied until a contraction of

about 15 mm. has occurred. When the rise in the manometer has been noted (it takes five to ten minutes to become steady), the capsule is broken by taking the tube between the finger and thumb at T and moving it quickly up and down. The final contraction may take as long as thirty or forty minutes, and for this reason it is best to have the apparatus set up and ready to run for a class, but the results have been found to be reliable if the following precautions are taken.

(1) The jar must be kept quite full of melting ice

(2) The *copper-covered* wire from disused electric-light bulbs has proved the most satisfactory contact for sealing into glass. A dab of sealing-wax on the outside of J was usually added as a precaution.

(3) See that the turpentine capsule does not roll down into the hot part of the tube when sealing it off, or an explosion may occur

Difficulty has sometimes been experienced owing to turpentine (or cinnamon oil) apparently absorbing oxygen slowly. However, with redistilled liquid and using ice, the results have been very satisfactory, this secondary absorption being then very slow.

### HYDROGEN SULPHIDE

#### 68. HOME-MADE KIPP'S APPARATUS

*J. H. H. MacRae and T. T. Richards*

Ferrous sulphide is contained in a combustion tube (length 5 in., diam. 1 in.), which is packed to the depth of  $\frac{1}{4}$ -in. with broken glass, and stands in a flask of 800 c.c. capacity. The flask is placed on

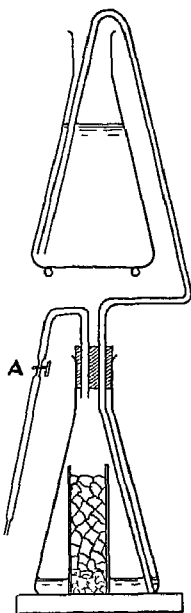


FIG. 40



the base of an iron stand. A similar flask, containing hydrochloric acid, stands on a retort ring. The glass tubing used is of  $\frac{1}{4}$ -in. diameter. The siphon is started by connecting the rubber tubing A to a water-pump, and afterwards works automatically.

The advantages of this apparatus are :

1. It is inexpensive and consists of parts always in stock and readily replaceable.

- 2 Its erection provides a Sixth-Form pupil with a good exercise in manipulation.

## 69. HYDROGEN SULPHIDE APPARATUS

*A. Eslick and S. V. Brown*

The apparatus is illustrated in side view in Fig. 41. Sulphuretted hydrogen is generated in the usual manner by the action of hydrochloric acid on ferrous sulphide in the generator (G), a tall glass tube packed with the solid. Acid is dropped on to the solid from the jet (J), a few drops at a time, and trickles down the generator, to reach eventually the outlet tube (T), and thence the gas jar (S) which is filled with water and spent acid ; as spent acid collects in S, it spills over into the large (1,000 c.c.) beaker (B), which is emptied from time to time

As the gas-pressure rises (assuming that the gas-outlet tube (D) is dipped into a test-tube full of liquid) the water-level in T is depressed to balance this pressure, and therefore the depth to which T is immersed in S must be a little greater than the length of a test-tube. The outlet tube (D) is not sealed in any way after use, since the fairly rapid solubility of the gas in the liquid in T tends to cause this liquid to be sucked up into the generator.

The acid, controlled by the Mohr clip (M), is delivered under pressure from the large bottle shown, which is placed on a shelf above the apparatus, the height of the bottle should be about 12 in. above the jet (J) so

that the pressure at which the acid is delivered will be in excess of any gas-pressure in the generator (G).

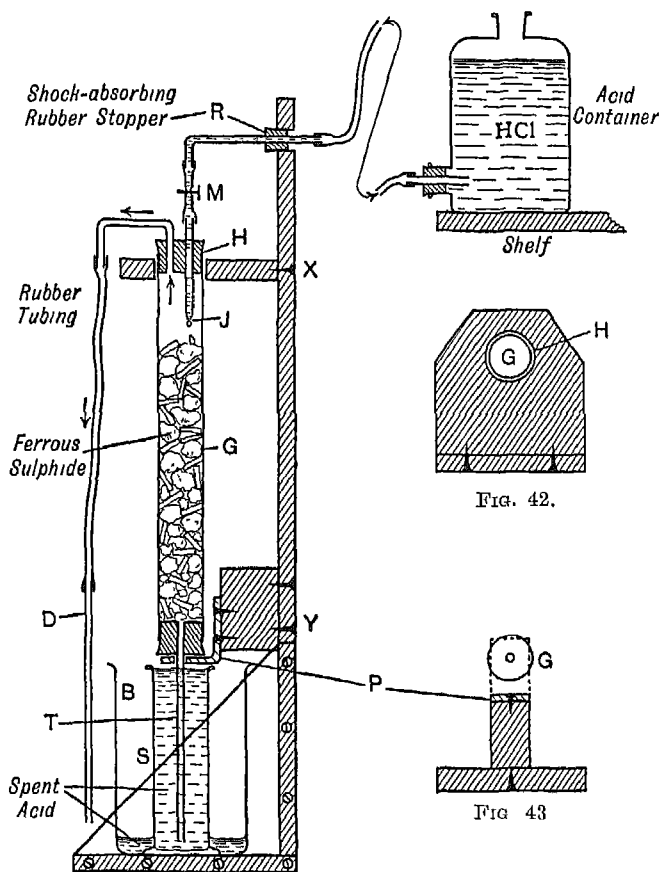


FIG. 41.

FIG. 42.

FIG. 43.

The advantages of this apparatus over the ordinary Kipp are :

(1) The acid is entirely used up, whereas in the Kipp the acid has to be replaced when only partly used because of the slow rate of gas delivery.

(ii) More solid and acid can be stored in the apparatus than in the Kipp ; hence recharging is less frequent.

(iii) The rate of gas-delivery remains constant to the last drop of acid, except in so far as it is reduced by the very gradual decrease in surface-area of ferrous sulphide.

(iv) In the unlikely event of the clip (M) allowing the acid to flow, uncontrolled, over the ferrous sulphide, the whole acid supply passes right through the apparatus in a few minutes, and only a comparatively small quantity of gas is generated ; in the case of a similar accident, the Kipp would continue to produce gas until all the materials were used up.

There are a few points worthy of note in the construction of the wooden stand. Fig. 42 is a horizontal section of the apparatus at the level X in Fig. 41. Fig. 43 is a section at level Y. In assembling the apparatus, the generator tube is lowered through the hole (H) (a sliding fit) and the tube (T) passed through the small hole in the bent brass strip (P), until the weight of the generator is taken by P *via* the rubber stopper ; this arrangement ensures that this rubber stopper cannot fall out of the generator.

## 70. DISTRIBUTION OF HYDROGEN SULPHIDE

*J. Lambert*

The following idea has been found useful in a rather large class with limited fume-chamber accommodation. If, as in group 2, it is sometimes necessary for sulphuretted hydrogen to be passed for some few minutes into the solutions of several pupils, two or three Kipp's apparatus are totally inadequate to meet the case. Furthermore, the laboratory speedily becomes almost unbearable.

This apparatus, which can easily be made in the wood-work department, multiples fourfold the number of pupils which can be supplied, and at the same time reduces to a minimum the amount of gas which escapes.

The holes in the top board are made to fit tightly the

rubber corks, which in turn fit the ordinary  $6 \times 1$  boiling tube. The corks are firmly fixed into the board with seccotine, and about  $\frac{1}{4}$ -in. clearance is allowed between the bottom of the boiling tubes and the bottom board.

*The Procedure.*—The corks are first fitted with standard  $6 \times 1$  boiling tubes which are empty. The pupil brings along his own boiling tube containing his solution, and a small piece of glass tubing. He fixes the latter in the rubber tubing just below the cork and fits on his boiling tube, placing the empty one near at hand. On removing

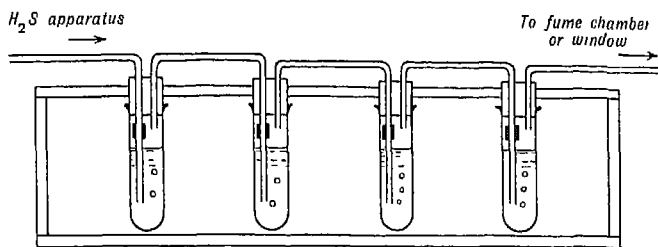


FIG. 44

his own tube he inverts the order, taking away his tube and piece of glass tubing and replacing the original empty boiling tube. In this way the supply of the gas can be tapped off at any one of the four points. The operation becomes almost automatic after one or two lessons.

The limit to the number of tubes which it is possible to employ depends only on the pressure which the sulphuretted hydrogen apparatus generates, the required pressure being the sum of the four respective depths which the inlet tubes are below the surface of the liquid in each of the tubes.

## 71. DEVICE TO KEEP DOWN THE SMELL

*H. G. F. Micklewright*

The simple device indicated in Fig. 45 has been found effective in reducing the odour which accompanies the

use of this gas in analysis. The following train of apparatus is fitted to the "Kipp" or other source of the gas: wash-bottle, small conical flask for precipitation, tap or Mohr clip, a large jar filled with moist sawdust and slaked lime (*vide Science Masters' Book*, Series I, Pt. 2, p. 106), filter-pump. The tap being turned on, the air is displaced from the flask by passing a rapid stream of gas for a few

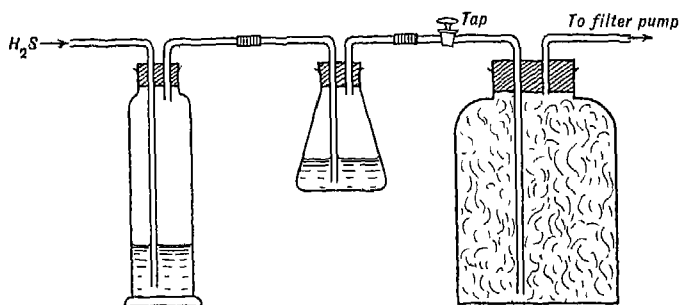


FIG. 45.

seconds. The tap is then turned off and the flask is shaken so as to keep the liquid saturated. When precipitation is deemed complete, the gas supply is turned off, the pump started, the tap between the flask and the jar turned on, and the flask detached from the wash-bottle.

### NITRIC ACID

#### 72. DECOMPOSITION OF NITRIC ACID BY HEAT

*D. Healy*

To illustrate the decomposition of nitric acid by heat, the following modification of the usual experiment may be carried out.

Instead of the churchwarden, a piece of hard glass or transparent silica tubing,  $\frac{1}{2}$ -in.- $\frac{3}{4}$ -in. bore, is used. One end is drawn out and suitably bent, and into the other is

sealed, by a plug of plaster of Paris, an ordinary clay pipe, with about a 2-in. stem.

The tube, held in a slanting position with the narrow end in a trough of water, is gently heated by means of a Ramsey burner, and nitric acid is poured slowly into the pipe. The brown nitrogen peroxide is seen clearly in the hot tube, and the oxygen may be collected in the usual way over water.

Since the temperature required to decompose nitric acid is not high, there is very little risk of the tube breaking—a risk which is eliminated if silica tubing is employed. Instead of the short clay pipe, a small dropping funnel, with the tail bent at about  $120^\circ$ , may be used.

### PREPARATIONS—INORGANIC

#### 73. BARIUM SULPHITE FREE FROM SULPHATE

*R. D. Reid*

Practically all specimens of sodium sulphite solution contain a proportion of sulphate, and it is therefore difficult to demonstrate the fact that the precipitate obtained with barium chloride is completely soluble in hydrochloric acid. The following is a very obvious way out of the difficulty. A few drops of alkali in half a test-tube of water are neutralized by sulphur dioxide from the "siphon." The precipitate then formed with barium chloride is readily soluble in dilute hydrochloric acid

#### 74. CERIUM COMPOUNDS FROM LIGHTER "FLINTS"

*A. J. Mee*

Lighter "flints" contain cerium alloyed with iron and form a good source of cerium compounds, particularly for class demonstration.

Cerium is soluble in dilute sulphuric acid ; hence, when the " flint " is dissolved in dilute sulphuric acid, a mixture of ferrous and cerous sulphates is formed. If a saturated solution of potassium sulphate is added to this solution, after filtering from carbon, a white crystalline precipitate of the double cerous potassium sulphate is thrown down. This salt,  $\text{Ce}_2(\text{SO}_4)_3, \text{K}_2\text{SO}_4, 2\text{H}_2\text{O}$ , is insoluble in a saturated solution of potassium sulphate, and is filtered off. Half a dozen of the ordinary small flints will provide sufficient of this salt to demonstrate the properties of cerium compounds.

The following experiments are suggested .

1 Prepare cerous oxalate by dissolving a little of the double sulphate in hot water and adding a solution of ammonium oxalate or oxalic acid. The white precipitate of cerous oxalate is at first curdy, but later turns crystalline. It should be filtered off, and if there is sufficient of it, it may be dried and ignited, when cerium dioxide,  $\text{CeO}_2$ , is formed as a faint yellow powder.

2. Prepare cerous hydroxide by adding sodium hydroxide to a solution of the double sulphate in water. It separates as a white precipitate, which, however, gradually changes in colour through red to yellow, being oxidized by the air to ceric hydroxide. The oxidation may be brought about more rapidly by adding a little sodium hypochlorite to the precipitate.

Other experiments can easily be devised by consulting the text-books.

## 75. IODINE FROM RESIDUES

*G. Fowles*

Almost fill a large enamelled basin with iodine residues. Make the liquid strongly alkaline—or iodine will be lost by evaporation—using waste caustic soda if available ; otherwise use solid caustic soda or washing soda. All the free iodine is thus fixed as sodium iodide and any cuprous iodide (from the volumetric work) is converted into

sodium iodide. Evaporate to about a quarter of the original volume. The highly soluble sodium iodide will be in the liquid. Decant this liquid into a large (700 c.c.) flask; wash the solid residue once with a small quantity of water and throw the solid away. Strongly acidify the liquid with concentrated hydrochloric acid; any sodium iodate now yields up its iodine;  $\text{HIO}_3 + 5\text{HI} = 3\text{I}_2 + 3\text{H}_2\text{O}$ . Dissolve a teaspoonful of sodium nitrite in about 50 c.c. of water and add this in small quantities to the liquid, agitating the flask and cooling if necessary, until no more iodine is precipitated (HCl and HBr are unaffected by nitrous acid). To decide this point, allow the solid to settle, take a trial portion of the liquid and see if the addition of acid or nitrite will precipitate any more iodine; more acid is often required, for sodium nitrite, like sodium carbonate, neutralizes the acid



Let the flask stand for some hours until all the iodine has settled, then decant and throw away the liquid, wherein are most of the impurities. Now swirl the crude iodine into a retort, the stem of which should not be too narrow, and add water so that the retort is about half-full. On applying heat the whole of the iodine comes over with the steam in a few minutes. The iodine should be collected in a flask half-filled with cold water and standing in a basin of water. The stem of the retort is inserted in the neck of the flask, but the end of the stem is kept just clear of the water; thus it is kept hot by the steam so that there is little likelihood of its getting choked with iodine. The flask should be rotated while the iodine is coming over. The receiver contains practically pure iodine and water. Allow the iodine to settle, then, as thoroughly as possible, drain off the iodine water, which may be discarded. Leave the flask, loosely stoppered, in a warm cupboard until the iodine is somewhat dry. In this condition it is quite suitable for several purposes, e.g. for the preparation of potassium iodide, or iodate, or of an iodine solution



for iodimetry If, however, a perfectly dry product is required, the iodine should be mixed, in small quantities, with twice its bulk of quicklime and sublimed.

## 76. IODINE FROM SEA-WEED

*Audrey H. Heap*

Previous efforts to extract the element in the school laboratory afforded a meagre yield and were unconvincing. By applying to the ashed weed the method of obtaining iodine from iodine residues outlined in *School Science Review*, vol. xiv, No. 56, June, 1933, iodine in appreciable quantities, readily demonstrable to a class, have been easily obtained

All sea-weeds do not contain the same percentage of iodine: those giving the greatest yield are the species of *Laminaria*, found in the drift kelp. The wracks, which can be gathered on most coasts, contain considerably less iodine.

<i>Laminaria digitata</i>	.	0 45	per cent. of dry weed	} drift kelp.
<i>Laminaria stenophylla</i>	.	0 48	" " " "	
<i>Fucus serratus</i>	.	0 086	" " " "	} wracks.
<i>Fucus vesiculosus</i>	.	0 03	" " " "	

We collected 5 to 6 lb. of the moist drift kelp (popularly called ribbon sea-weed) from the shore after a rough sea. The weed was lightly washed, but no attempt was made to remove the silicious skeletons adhering tenaciously to the weed. The kelp was air-dried and the product crushed and charred between sand trays, thus rendering reduction of its bulk, by powdering, a possibility. The powder was heated in a large crucible until practically free from carbon. The ash so obtained was lixiviated with water, filtered, and the filtrate reduced to small bulk. After treatment with hydrochloric acid and sodium nitrite and distilling, violet vapour, condensing in a test-tube

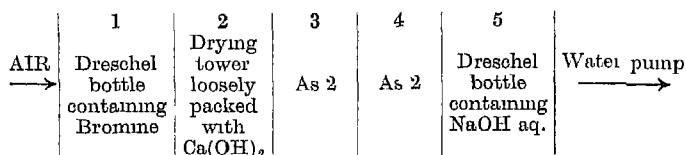
receiver as solid iodine, filled the flask. With a poorer sample of weed the violet coloration was observed and a straw-coloured distillate, giving a good test with starch solution, was produced.

By the dry distillation of a small portion of the weed, an unsaturated hydrocarbon and acid distillate were obtained. This was of interest since on an industrial scale such valuable products as acetone and the ethyl esters of some of the lower fatty acids have been obtained by the dry distillation of the gigantic *Macrocystis*, reaped from the ocean bed by submarine cutters. In another process inoculation of the wet weed with appropriate bacteria is employed. Fermentation is allowed to proceed and the neutralized liquid is distilled.

In addition to its importance as a source of iodine, the weed is a valuable fertilizer. In Jersey and Cornwall, where land is intensively cropped, where straw and artificial manures must be imported, use is made of the seaweed thrown up by Atlantic breakers. This is collected, strewn upon the ground, allowed partly to decay during the winter and ploughed in before potato planting.

## 77. BROMINE BLEACHING POWDER

*E. J. Williams.*



Bromine bleaching powder may be prepared by the arrangement of apparatus outlined above. From time to time, aspiration should be stopped and the lime in the

towers stirred with a glass rod. When Tower 2 is saturated, remove it and connect 3 to 1, if desired, adding a tower with fresh slaked lime between 4 and 5.

The substance is a brick-red powder, and some idea of saturation may be obtained from uniformity of the colour.

Bromoform may be obtained by distilling it with acetone as in the preparation of chloroform. It always seems to fall lower than ordinary bleaching powder as regards "available bromine," and many of its properties seem to merit further investigation if they have not previously been described.

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" " " **19**, 296.  
" " " **30**, 295 (1883)  
Lowig, *Ann. Chim. Phys* [3], **32**, 337  
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### 78. WHITE PHOSPHORUS FROM BONES

*G. H. Locket*

Although bones are no longer a commercial source of phosphorus (vide *School Science Review*, [58], Dec., 1933, p. 156), the following preparation is instructive and is now regularly carried out by boys.

The bones are best calcined in a muffle, or in a central-heating furnace, without first breaking them up. About 300 gm. of ash, finely powdered, are added in small quantities at a time to 320 gm (175 c.c.) of sulphuric acid, diluted with its own volume of water. The mixture is heated, with occasional stirring, on a water-bath for an hour or two (water being added, if necessary), and is then filtered, after cooling, at the pump, the solid being washed with about  $1\frac{1}{2}$  litres of water. The filtrate is evaporated (calcium sulphate which separates can be filtered off)

and when it is of a syrupy consistency, powdered charcoal is stirred in and the mixture worked up with a rod into balls, which harden on cooling and constitute the charge for the retort.

The retort is made from a piece of standard 2 in. gas tubing, 5 in. long, with threads on to which screw two caps; one of these is fitted with a nut for opening the retort, the other carries a reducing socket with a piece of  $\frac{5}{8}$ -in. tubing, bent at right-angles (the work was done by a blacksmith).

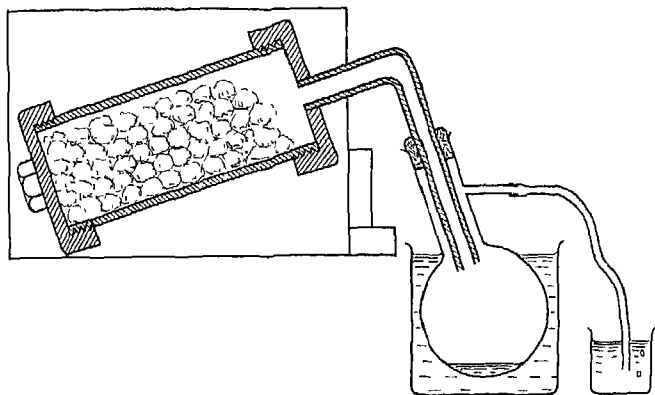


FIG. 46.

When the retort is loosely packed, the threads are covered with a paste of litharge and lead carbonate mixed with linseed oil, and the ends are screwed on. The retort should be left for a day and then placed in a muffle, the delivery tube passing into a pyrex distilling flask through a plug made of asbestos, soaked in phosphoric acid and covered with zinc oxide. A rubber tube from the side tube of the flask leads under water to form a seal.

During distillation, the receiver is immersed in cold water, which is allowed to get hot and is not changed. Reducing gases and steam soon come over, and the temperature is slowly raised as high as possible, the distillation taking two and a half to three hours.

The end of the retort should be removed as soon as possible after use. If it is warmed on a gas-ring, it can be shifted quite easily with a spanner and a wooden mallet.

The yellow phosphorus is left with dilute chromic acid for a day and then, after washing and melting, is drawn through a warmed tube, fitted with a glass-wool filter, into a test tube with water. Usually a perfectly white product is obtained. Yields about 15–20 per cent. of theory.

## 79. MONOCLINIC SULPHUR

*R. R. Finney*

It is quite unnecessary to use the large clay pot to prepare a specimen of monoclinic sulphur. The following method works well and is suitable for individual work; the older way is essentially one for demonstration purposes.

Carefully melt half a test-tube of sulphur, avoiding dark brown patches. The result should be a lemon-coloured liquid. Fold a filter paper, open as usual, but keep the paper dry. Hold it at the upper edge so that the filter paper retains its conical shape. Quickly pour the molten sulphur into the filter paper. When the crust completely covers the molten sulphur, pierce the crust as usual and open out the filter paper flat on the bench. A fine collection of monoclinic needles is readily available for individual observation.

## 80. EXPERIMENTS WITH SULPHUR

*R. D. Read*

Class experiments with sulphur tend to be somewhat difficult and messy, but the following simple procedure demonstrates some of the important properties of the element. Put some flowers in a crucible lid or piece of

metal. Heat a piece of glass tubing until just not red hot. Dip the tube in the flowers. They melt, adhere, and burn on the end of the tubing. The fire can be replenished by further dipping. Some of the vapour passes up the inside of the tube and can be observed condensing as a liquid and in the form of flowers. By this means the quantity of sulphur dioxide liberated into the atmosphere is small, but it can be smelt by the experimenter, and the colour of a filter paper dipped in permanganate solution and held over the flame is at once destroyed.

### 81. URANYL ACETATE FROM RESIDUES

*A. J. Mee*

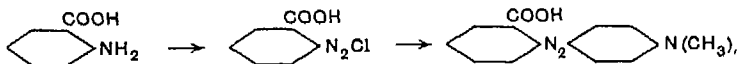
The liquid containing the uranium residues is treated with ammonium chloride and then made strongly alkaline with ammonia. It is then heated on the water-bath until the uranium separates as ammonium uranate. The precipitate is filtered off and dried at  $300^{\circ}$ . To obtain uranyl acetate, 100 gm. of the uranate are suspended in 600 c.c. of water, heated to  $80^{\circ}$ , and 50 c.c. glacial acetic acid are added. The solution is then evaporated until its bulk reaches 250 c.c. when, on cooling to room temperature, uranyl acetate,  $(\text{CH}_3\text{COO})_2\text{UO}_2 \cdot 2\text{H}_2\text{O}$ , crystallizes.

### PREPARATIONS—ORGANIC

#### 82. METHYL RED

*G. Fowles*

This useful indicator is prepared by coupling dimethylaniline with anthranilic acid.



This preparation of methyl red is based on that of T. F. Winnill, (*Jour. Chem. Soc.*, 1910, 2485). It works well.

Dissolve 5 gm. of anthranilic acid in a mixture of 150 c.c. of water and 5 c.c. of concentrated hydrochloric acid. Diazotize the solution at room temperature by adding, in small portions, 2.5 gm. of sodium nitrite. Allow the solution to stand for half an hour. Meanwhile, prepare a solution of 4.65 gm. of freshly-distilled dimethylaniline in 50 c.c. of water, acidified with 5 c.c. of concentrated hydrochloric acid. Pour the diazonium salt into this solution and add 50 gm. of sodium acetate. Warm to 40° C., when the red dye quickly separates. Allow it to stand for three hours to complete the reaction. Filter, and wash with water—in which the dye is highly insoluble. The produce is best air-dried; it is rather difficult to crystallize. To prepare a stock solution for use as an indicator in acidimetry, dissolve about 0.2 gm. of the dye in a litre of alcohol (industrial spirit serves quite well).

### 83. PHENACETIN—SYNTHESIS FROM BENZENE AND ALCOHOL<sup>1</sup>

#### NITROBENZENE<sup>2</sup>

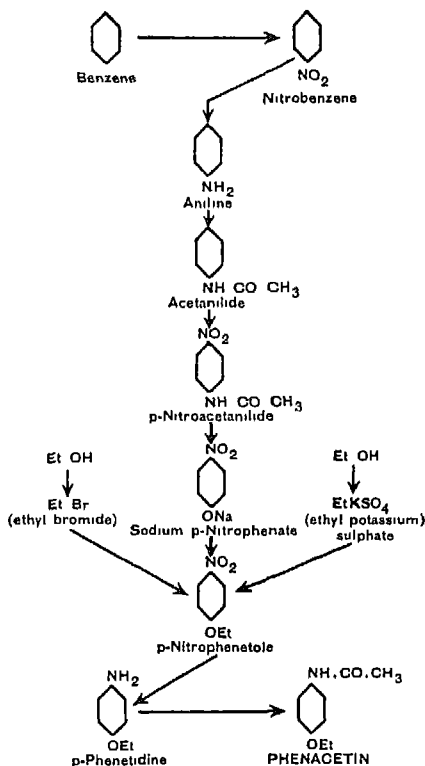
Benzene	.	.	.	.	.	50 gm.
Nitric acid (conc)	.	.	.	.	.	80 "
Sulphuric acid (conc)	.	.	.	.	.	120 "

The well-cooled mixture of the two acids is added slowly from a tap funnel to the benzene, which is contained in a 500 c.c. flask; the contents of the flask are well shaken after each fresh addition. Nitrous fumes are evolved and a considerable amount of heat is developed. Care must be taken that the temperature

<sup>1</sup> In the early days of the European War, a process for making phenacetin was worked out in the organic laboratories of the Imperial College of Science. Professor Thorpe (see *School Science Review*, vol. xiii, no. 51) suggests this as a revision course of organic chemistry suitable for chemistry specialists. Dr. M. A. Whiteley has kindly supplied the following from records of the processes used in 1914.—Ed.

<sup>2</sup> The preparations of nitrobenzene, aniline and acetanilide have been included to make the account of the synthesis complete in itself. The working details for these preparations will be found to be more precise than those usually given and somewhat different.

does not exceed  $50^{\circ}$ – $60^{\circ}$  by immersing the flask, if necessary, in cold water. The nitrobenzene separates as a brown oily layer on the surface of the acid liquid. When the acid has all been added, an operation which lasts about half an hour, the mixture is heated for about twenty minutes in a water-bath at  $60^{\circ}$ , and again well



shaken. The contents of the flask, on cooling, are poured into a stoppered separating-funnel, the lower layer of acid is removed, and the nitrobenzene washed with 50 c.c. of water, which removes most of the remaining acid. The lower oily layer of nitrobenzene is then transferred to a litre flask, containing a dilute solution of sodium carbonate, and steam distilled; this opera-



tion removes all impurities of acids, nitrophenols and dinitrobenzene from the nitrobenzene, which is obtained as a pale yellow heavy oil in the distillate ; it is separated as completely as possible from the water, and dried by shaking with successive small quantities of calcium chloride until the liquid is clear. The yellow liquid is decanted or filtered from the calcium chloride, and distilled from a distilling flask, using an air condenser. At first a little benzene distils over ; the temperature then rises and the nitrobenzene, which has b p.  $210^{\circ}$ , can be collected at  $208^{\circ}$ – $212^{\circ}$ . Yield 60 gm.

#### ANILINE AND ACETANILIDE

Nitrobenzene	.	.	.	50 gm.
Tin, granulated	.	.	.	90 "
Hydrochloric acid	.	.	.	170 "

The tin and nitrobenzene are introduced into a round flask (1.5 litres), provided with a long reflux air condenser, and the mixture is heated for a few minutes on the steam-bath. The flask is then removed and the concentrated hydrochloric acid added in quantities of 5 to 10 c.c. at a time, the mixture being shaken repeatedly. The liquid should become hot and boil quietly, but if the action becomes too violent, it must be moderated by cooling the flask in cold water. The addition of the acid should take about thirty to forty minutes ; the flask is then replaced on the water-bath without the air condenser and heated until no odour of nitrobenzene can be detected. The contents of the flask are then steam distilled, to remove the last traces of the nitrobenzene, and while still warm, a solution of sodium hydroxide (140 gm. in 200 c.c. of water) is added until the stannic oxide, which is at first precipitated, nearly redissolves and the liquid is strongly alkaline. Should the mixture begin to boil during the addition of the alkali, it must be cooled. The aniline, which separates as a dark oil, is removed by distillation in steam, the distillate being collected in a *clean* separating funnel, and the operation

being continued so long as any oil appears in the distillate. The aniline, which separates as a heavy oil in a *clean* separating funnel, is then separated from the aqueous layer (aniline water), dried over a few pellets of potassium hydroxide, and distilled at 182°–183°. Yield about 30 gm

The residue of aniline left in the distilling flask, and that dissolved in the aniline water, can be recovered as acetanilide by rendering the mixture slightly acid with hydrochloric acid, adding a slight excess of sodium acetate, followed by a small excess of acetic anhydride, and shaking the mixture vigorously in a closed flask until the acetanilide is precipitated in crystalline form. After standing for about an hour, the acetanilide is collected on a filter and crystallized from boiling water; it forms rhombic plates, m.p. 115°. Yield 9 gm

*p*-NITROACETANILIDE

Acetanilide . . . . .	50 gm
Sulphuric acid (conc.) . . . . .	84 c.c. and 15 c.c.
Nitric acid (conc) . . . . .	22 c.c.

50 gm. of acetanilide are dissolved in 84 c.c. of concentrated sulphuric acid, keeping the temperature below 40°. A cooled mixture of 22 c.c. of concentrated nitric acid with 15 c.c. of concentrated sulphuric acid is slowly dropped in with stirring, while the temperature is maintained below 15°. When the addition is finished, the mixture is allowed to remain, with occasional stirring, for about half an hour, and is then poured into a litre of iced water: the precipitate is collected immediately on cloth, washed several times with water, and finally with sodium acetate solution until neutral to congo red paper. It is then drained by squeezing and the damp cake weighed. A small weighed quantity is dried and recrystallized for a specimen; the remainder of the wet cake is used in the following preparation. The pure compound melts at 210°–211°.

SODIUM *p*-NITROPHENATE

<i>p</i> -Nitroacetanilide . . . . .	50 gm.
Sodium hydroxide . . . . .	35 "
Water . . . . .	200 c.c.

The damp nitro compound (equiv. 50 gm) is mixed with the sodium hydroxide solution in a round-bottom flask, and boiled on a sand-bath; no reflux is used, fresh water being added when necessary<sup>1</sup>. After fifteen to twenty hours, the steam becomes neutral and the contents of the flask are allowed to cool, when the solid is collected and recrystallized from water, using a hot-water funnel. The mother liquors obtained in the recrystallization, when evaporated to about one quarter of their bulk, yield a further quantity of crystals. The yellow crystalline sodium salt is converted, by drying at 100°, into the red anhydrous salt. Yield about 44 gm.

*p*-NITROPHENETOLE

Sodium <i>p</i> -Nitrophenate . . . . .	25 gm
Ethyl potassium sulphate . . . . .	50 "
Glycerol . . . . .	25-30 c.c.

The glycerol is dehydrated by heating it for twenty minutes at 180° and is then allowed to cool. The anhydrous sodium *p*-nitrophenate and the potassium ethyl sulphate are rubbed up with the glycerol in a mortar to a paste, which is transferred to a small round flask fitted with an air condenser; the mixture is heated in a wax bath at 175° until free refluxing takes place (about eighty minutes). On cooling, the *p*-nitrophenetole solidifies at the surface in a dark-coloured cake. The product is treated with 300 c. c. of water, cooled in ice and filtered. The solid residue is melted under dilute sodium hydroxide solution, cooled and filtered as before, and after drying, it is crystallized from petroleum (b.p. 60°-80°). From

<sup>1</sup> During this operation, the *o*-nitroacetanilide produced in the nitration of the acetanilide is converted to *o*-nitraniline, which is carried off in the steam. This should be collected by condensing the vapour so long as it is coloured yellow. The *o*-nitraniline crystallizes from this distillate and should be kept as a specimen.

the rapidly cooled solution, 20 gm. of a pale red crystalline material are obtained, which melts at  $50.3^{\circ}$ . A specimen is bleached by melting it under concentrated hydrochloric acid containing a little stannous chloride ; on recrystallizing the product from petroleum, a colourless sample of *p*-nitrophenetole is obtained, melting at  $58.4^{\circ}$ .

*p*-NITROPHENETOLE

Sodium <i>p</i> -Nitrophenate.	.	.	30 gm.
Ethyl bromide	.	.	23 "
Absolute alcohol	.	.	21 "

The anhydrous sodium *p*-nitrophenate is enclosed in a firmly-stoppered pressure bottle with the ethyl bromide and alcohol, and heated in a water-bath at  $100^{\circ}$  for seventeen hours. When cold, the contents of the bottle are transferred to a flask and steam distilled for half an hour, to remove alcohol and unchanged ethyl bromide. From the distillate the ethyl bromide is recovered by dilution with water. The residue is cooled, with continual shaking, and the solid nitrophenetole is filtered off, the filtrate being reserved for the recovery of sodium bromide. The crude brown-coloured nitrophenetole is purified by melting twice under dilute sodium hydroxide, then under water, and then under concentrated hydrochloric acid. Each time the melt is resolidified by shaking the flask under running water. Finally, the nitrophenetole is melted under concentrated hydrochloric acid containing a small amount of stannous chloride, and the product, now almost colourless, is filtered off, washed with water and air dried. Yield about 25 gm. A small sample is recrystallized ; m.p.  $59^{\circ}$ .

*Recovery of Sodium Bromide.*—The filtrate containing the sodium bromide is evaporated to dryness and charred upon iron trays. The residue is extracted with hot water, and the solution of sodium bromide thus obtained is freed from the small quantity of alkali present by neutralization with a 10 per cent. solution of hydrobromic acid. From the neutral solution the sodium

bromide may be obtained by evaporation, and used for the preparation of ethyl bromide.

### *p*-PHENETIDINE

<i>p</i> -Nitrophenetole . . . .	22.6 gm.
Methylated spirits . . . .	100 c.c. and 30 c.c.
Hydrochloric acid (conc.) . . . .	5 c.c. and 25 c.c.
Iron filings . . . .	25 gm.

The *p*-nitrophenetole, dissolved in 100 c.c. of methylated spirit, is placed in a 500-c.c. flask (round bottom) fitted with an air condenser. 5 c.c. of concentrated hydrochloric acid are added and the contents of the flask heated to boiling on the steam-bath. 23 gm. of iron filings are then added to the solution in four equal portions, at intervals of about five minutes, during which time the liquid is kept boiling regularly and fairly vigorously, to prevent caking of the iron; finally, the boiling is continued for a further two hours, any loss of spirit being made good by addition (about 30 c.c.). When the heating is completed, a methylated spirit solution, containing 4 gm. of sodium hydroxide, is added to the contents of the flask and the whole filtered through a Buchner funnel, the residue being washed with a little hot methylated spirit. The base is precipitated as its hydrochloride from the filtrate by addition of 25 c.c. of concentrated hydrochloric acid, and separated by filtration. A second crop of crystals can be obtained on evaporation and crystallization.

To estimate the weight of pure hydrochloride in the sample thus obtained, weighed portions are dissolved in absolute alcohol, filtered to remove any sodium chloride, and the filtrate evaporated to dryness and weighed. Yield 18 gm.

### PHENACETIN

The acetylation of the *p*-phenetidine to "phenacetin" is carried out in an exactly similar manner to the acetylation of aniline to acetanilide, q.v.

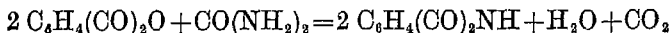
From 5 gm. of *p*-phenetidine hydrochloride, 4.5 gm.

of phenacetin are obtained. It is crystallized from benzene and melts at 135°-136°.

#### 84. PHTHALIMIDE

*G. Fowles*

In 1919 W. Herzog found that phthalimide could be easily prepared by the interaction of phthalic anhydride and urea. This discovery brings the synthesis of some interesting substances within the capacity of the advanced course pupil. Thus, beginning with naphthalene and proceeding through phthalic acid, phthalic anhydride and phthalimide anthranilic acid is reached by the oxidation of phthalimide. By coupling this acid with dimethylaniline, the valuable indicator methyl red is obtained. Alternatively, the anthranilic acid may be condensed with chloracetic acid and thus phenyl-glycine o-carboxylic acid prepared: this, fused with caustic potash affords indigotin, whose aqueous solution oxidized in the air yields indigo. Herzog's method works splendidly; the details are as follows: Phthalic anhydride (2 mols) mixed with urea (1 mol.) is heated in a long-necked flask—we use a boiling tube. The reaction begins at 130-135° and the temperature rises to 150° without further heating. At the end the liquid mass suddenly solidifies. When the tube is cold remove the product and wash it with water. The yield is 90 per cent. M.P. 231° C.



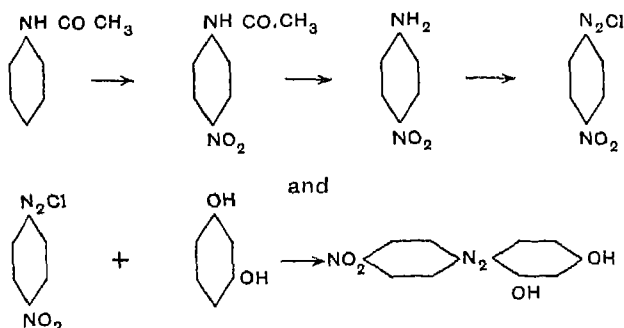
#### 85. RESORCIN AZO-*p*-NITROBENZENE

*G. Fowles*

The original paper by Suitzu and Okuma on the preparation of *p*-nitrobenzeneazoresorcinol is in Japanese, and the English abstract is not very helpful. Profiting by a statement by Ruigh in 1929, that the dye could be

prepared by coupling *p*-nitraniline with resorcinol, the following procedure was worked out

Using acetanilide as the starting material the following changes are involved .



The preparation of *p*-nitraniline appears in several books but the details have been given here for the sake of completeness.

Dissolve 5 gm. of finely-powdered acetanilide in 9 c.c. of concentrated sulphuric acid, keeping the solution below 40° C. Cool the solution in ice water to 5–10° C and add slowly a cooled mixture of 2.5 c.c. of concentrated nitric acid and 1.5 c.c. of concentrated sulphuric acid. Shake thoroughly during the addition and keep the temperature below 15°. Allow the mixture to stand for thirty minutes, then pour it into 200 c.c. of ice-cold water: *p*-nitroacetanilide is precipitated. Filter at the pump and wash with water. The product contains isomers which need not be removed; the pure substance melts at 211° C. Add the washed precipitate to a mixture of 10 c.c. of water and 5 c.c. of concentrated sulphuric acid and hydrolyse by warming until solution is effected. Now cool and add a solution of 8 gm. of caustic soda in 30 c.c. of water to neutralize the acid; *p*-nitraniline, with small amounts of accompanying isomers, is precipitated. Filter at the pump and recrystallize the product from a little hot water (M.P. = 147° C.). Take 2.5 gm. of

*p*-nitraniline and suspend it in a mixture of 5 c.c. of concentrated hydrochloric acid and 10 c.c. of water. Dissolve 2 gm. of sodium nitrite in 10 c.c. of water and diazotize in the usual way, keeping the solution below 15°. Now dissolve 2 gm. of caustic soda in about 100 c.c. of water and dissolve 3.5 gm. of resorcinol in this liquid. Slowly add the diazonium salt to the alkaline solution of resorcinol. The purple dye is immediately precipitated. (The supernatant liquid may be used forthwith as a test for magnesium.) It was found difficult to wash the alkaline form of the dye. However, on acidifying the liquid containing the precipitated dye with hydrochloric acid (in which the dye seems insoluble), a scarlet product is obtained which is comparatively easy to wash with water. The washing should be done immediately, otherwise the precipitate slowly changes in colour from scarlet to chocolate. The chocolate form gives the magnesium reaction, but is difficult to wash.

To prepare a solution of the reagent, dissolve about 0.1 gm. of the dye in 100 c.c. of dilute caustic soda solution, say 1-4 per cent., but any strength seems suitable. To carry out the test, add to the magnesium solution, which should be dilute and made alkaline with caustic soda, a few drops of the reagent; a blue colour immediately appears if the magnesium is dilute, a blue precipitate in more concentrated solutions. If too much of the reagent is added, the purple colour of the dye obscures the blue of the magnesium complex. Nickel, cobalt, aluminium and manganese interfere with the test, also a considerable concentration of ammonium salts. In our experience the concentration of ammonium salts used in ordinary group analysis does not prevent the formation of the blue precipitate.



## SAPONIFICATION

## 86. SAPONIFICATION OF OLIVE OIL

*W. R. Fearon*

The course and products of the alkaline hydrolysis of a typical lipid can be demonstrated conveniently by the following simple method :

Shake up 4 c.c. of olive oil with 4 c.c. of 20 per cent. sodium hydroxide. A white emulsion is formed, consisting of a disperse phase of oil in a continuous aqueous phase. Heat the mixture in boiling water. The emulsion is unstable in presence of the excess of alkali, and soon resolves into a layer of oil on a layer of water. Saponification occurs gradually at the interface, and is aided by shaking the tube from time to time.

After at least half an hour, remove and cool the tube. It now shows three layers : a lower one of glycerol and alkali, an upper one of unchanged oil, and an intermediate solid layer of soap (sodium oleate) which has been "salted-out" of the lower layer by the excess of alkali.

Carefully remove the liquid layers by decantation into a second tube, and show the presence of glycerol by means of the nitro-chromic test (Fearon and Mitchell, 1932, *Analyst*, 57 ; 372). Add excess (3-5 c.c.) of concentrated nitric acid and about 5 drops of 5 per cent. potassium chromate. A blue colour develops in the aqueous layer owing to the presence of the alcohol groups in the glycerol.

Rinse the small cake of soap with cold water, and then dissolve in about 10 c.c. of water, with the aid of heat. The solution shows the reactions characteristic of a soap.

(i) Add 1 c.c. of saturated brine to 3 c.c. solution.

The soap is "salted-out," and rises to the surface of the mixture.

- (ii) Add a few drops of a mineral acid to 3 c.c. solution. The soap is decomposed, and a white precipitate of oleic acid separates out.
- (iii) Add a few drops of 5 per cent. calcium chloride to 3 c.c. solution. A white precipitate of calcium oleate (lime soap) separates out.

## PHYSICAL CHEMISTRY

### ADSORPTION

#### 87. GLASS AN ADSORBENT

*G. Fowles*

Dissolve a small amount, say 0.1 gm., of methyl violet in a little warm water in a 500-c.c. flask. Rotate the flask so that the whole of the inside is wetted by the solution. If any specks of solid dye, hidden by the deep colour of the liquid, remain undissolved, they will adhere to the flask and spoil the experiment; swill them off and heat the liquid until they disappear. Cool the flask and pour away the solution. Rinse the flask two or three times with water or until the runnings are colourless: the glass still retains a violet sheen which water seems unable to remove. (Actually, a small fraction, depending on the adsorption equilibrium, is removed each time.) Now rinse the flask with 20 c.c. of alcohol or glacial acetic acid: a deep violet solution is obtained.

#### 88. ADSORPTION BY METALLIC HYDROXIDES

*G Fowles*

Completely precipitate the hydroxide from 6 gm. of alum and well wash the product with water. In a large flask place 200-300 c.c. of water and add an amount of congo red of the size of two rice grains. Warm and

swirl the flask so as to display the red liquid. Now add the aluminium hydroxide, boil for two or three minutes and filter. The filtrate runs through colourless, while the dye and aluminium hydroxide form a red jelly.

## 89. ADSORPTION INDICATORS

### *A. W. Wellings*

Two glass beakers, each containing 1 litre of distilled water, are placed side by side, and about three-milligrams of sodium eosinate is dissolved in each litre. At this degree of dilution the indicator is highly dissociated, and both the yellow colour and the green fluorescence are due to free eosin anions. If to one solution (the other serves for comparison) about 5 drops (0.05 c.c.) of  $N.AgNO_3$  are added from a micro-burette, no change in the colour of the eosin can be observed; apparently, in this great dilution, the sparingly soluble silver eosinate remains dissolved and dissociated. If now, to this solution, 1 drop of  $N.KBr$  be added from a second micro-burette, a strong deepening in colour occurs. the final tint is reddish, and it will also be noticed that the fluorescence decreases at the same time, though the solution remains quite transparent.

The explanation of these changes is as follows: silver bromide has been produced in a colloidal form with a very large degree of dispersion; also, as  $Ag^+$  ions are present in excess, some of them are adsorbed on the  $AgBr$ , to form, when the surface is saturated, the complex  $[AgBr]Ag^+$ . This positively charged body repels the remaining free  $Ag^+$  ions, but attracts the negatively charged eosin anions; and as a result of this deformation of the electron system of the dye anion, its colour and tendency to fluoresce is altered.

That this is so can be shown by continuing the titration. If 2 or 3 drops (i.e. a further 1 or 2 drops) of  $KBr$  are added, the red colour first of all deepens still

more, because the quantity of AgBr, and therefore also the quantity of adsorbed dye anion, is increased; then, with the addition of 4 drops of KBr, a distinct lightening in colour occurs, for the concentration of the excess  $\text{Ag}^+$  ions, and consequently the force exerted by the  $[\text{AgBr}]\text{Ag}^+$  body, are both decreased: thus the adsorption of the eosin is diminished. With the addition of 5 drops, the equivalent point is reached and the original yellow colour and fluorescence of the eosin anions are restored, the degree of this may be enhanced when the equivalent point is passed by the addition of 6 drops of KBr. The equivalent body is now  $[\text{AgBr}]\text{Br}^-$  since the eosin anions have been displaced from its surface by the excess of  $\text{Br}^-$  ions.

The above "colour game" can now be repeated as often as one likes by the alternate additions of  $\text{AgNO}_3$  and KBr, i.e. by the alternate attraction or displacement of the dye anions to or from the surface of the *equivalent body* (this is the term used by Fajans to denote the adsorbent at the exact equivalent point).

If the quantity of AgBr be increased by the addition of greater amounts of  $\text{AgNO}_3$  and KBr, the sol gradually becomes turbid, but the above colour changes can still be observed, not only in the larger quantities of sol, but also on the fine particles of AgBr.

### CATALYSIS

#### 90. RAPID ILLUSTRATION OF THE CATALYTIC OXIDATION OF AMMONIA AND A DEMONSTRATION OF CATALYSIS BY PLATINUM

*E. J. Williams and L. T. Taylor*

Into a conical flask put a small quantity of ammonium hydroxide (0.880). By means of a glass rod test this by addition to a solution of diphenylamine in concentrated sulphuric acid (0.05 gm. in 100 c.c.). No coloration shows absence of nitrates and nitrites.

Wind about 1 ft of platinum wire into a loose spiral round a pencil, suspend it from a piece of glass rod, heat it to redness in a Bunsen flame, and quickly introduce it into the flask. It will continue to glow, then get dull again, then reglow if more oxygen is added. This may be done by sending a puff of air into the flask by a blow-ball. Fumes of ammonium nitrate develop in the flask.

If, after a short time, the glass rod is again dipped into the ammonia, and then touched on the diphenylamine, a blue colour will prove the formation of nitrate or nitrite.

If the ammonium hydroxide is slightly warmed and oxygen itself slowly bubbled through, the platinum glow is increasingly striking, and the oxidation takes place with (quite safe but impressive) explosive violence.

## 91. OXIDATION OF AMMONIA TO NITRIC ACID

### *D Nealy*

The following apparatus is very suitable for demonstrating the manufacture of Nitric Acid from Ammonia.

Dreschel bottle, containing Ammonia solution + litmus.	Hard glass tube containing Platinized Asbestos	Dreschel bottle containing water + blue litmus.	Water pump.
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Air is drawn through the ammonia solution and the ammonia-air mixture passes over the heated platinized asbestos. The products then bubble into the water in the second Dreschel bottle. Sufficient nitric acid is produced in a few minutes to redden the litmus.

The Ammonia solution should consist of approximately equal volumes of 0.88 ammonia and water: if concentrated ammonia is used, the oxidation may be incomplete, and excess of ammonia will then pass into the second wash bottle. In any case white fumes of ammonium nitrate are generally present.

Oxygen should *not* be used instead of air, for with

oxygen the reaction is altogether too vigorous and a series of explosions occurs in the heated tube.

### CHEMICAL CHANGE—RATE OF

## 92 VELOCITY OF REACTION EXPERIMENTS WITHOUT A THERMOSTAT

*H. C. Palmer*

It is suggested that the experiment be done communally, using a very large volume of reaction mixture, say, 5 litres. When all is ready, the reaction is started and a stop-clock switched on at the same instant. Thereafter the members of the form visit the large bottle and withdraw portions for analysis. The change of temperature of a large volume is very slight, and by collating the form's results on a time titration graph, it is possible to draw a smooth curve and so obtain "ideal" titrations at fixed intervals on which to base the calculation constants.

This method has worked well with the hydrolysis of esters, the saponification of esters, the oxidation of formic acid by bromine and the reaction between sodium thio-sulphate and bromacetic ester.

## 93. CONDITIONS AFFECTING THE RATE OF CHEMICAL CHANGE

*E. R. Thomas and J. M. Burnett*

Experimental details are given in *The Science Masters' Book*, First Series, part 2, pp. 112-16. The curves in Fig 47 were obtained by the method there described.

Curve A represents the rate of reaction of dimethyl aniline; B, methyl-N-propyl aniline; C, methyl-isopropyl aniline, and D, di-ethyl aniline. The space-filling of the ethyl groups is shown here.

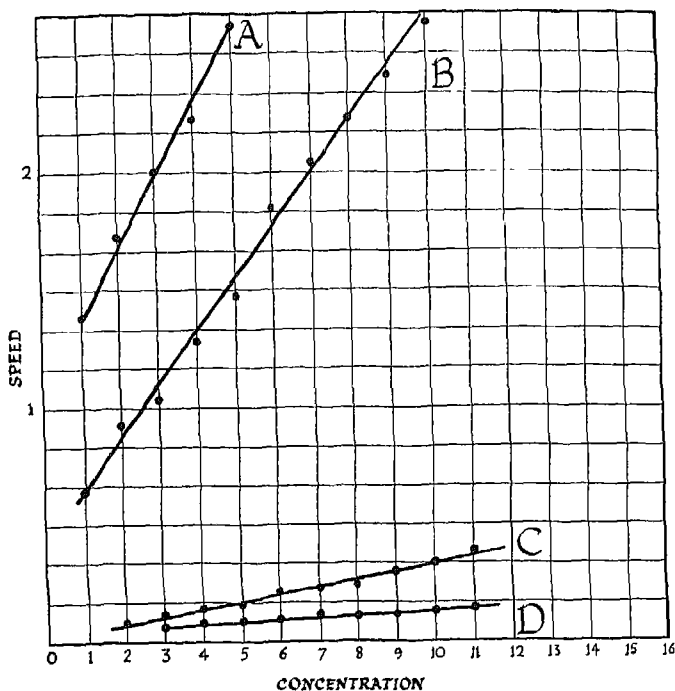


FIG 47.

#### 94. STATE OF CARBON DIOXIDE IN AQUEOUS SOLUTION

*E. J. Bowen*

In an aqueous solution of carbon dioxide, a very small fraction only exists as carbonic acid (molecules and ions), the remainder being merely dissolved carbon dioxide. If a little alkali is added, the carbonic acid is instantly neutralized and the solution becomes alkaline, but the disturbance of the equilibrium:  $\text{CO}_2 + \text{H}_2\text{O} \rightleftharpoons \text{H}_2\text{CO}_3$  leads to the production in a second or so of more carbonic acid, and the solution, if the alkali is less than equivalent

to all the carbon dioxide, quickly becomes acid again. This rapid reaction can be demonstrated by a simple modification of a Hartridge-Roughton tube, as shown in the diagram. Two large funnels or other reservoirs are connected by rubber tubes to the Y-shaped capillary tube (of about 2 mm bore)

This tube can be arranged either vertically or horizontally. One funnel is filled with N/1000 sodium hydroxide solution, and the other with water saturated with carbon dioxide, to which 50 c. c. of phenol phthalein solution or more per litre have been added. After removal by squeezing of air trapped in the rubber tubes, the clips are opened so that the solutions flow down to mix at the junction of the Y. Here all the carbonic acid is neutralized, as shown by the pink colour of the indicator. During the passage of the liquid through the capillary tube, however, more

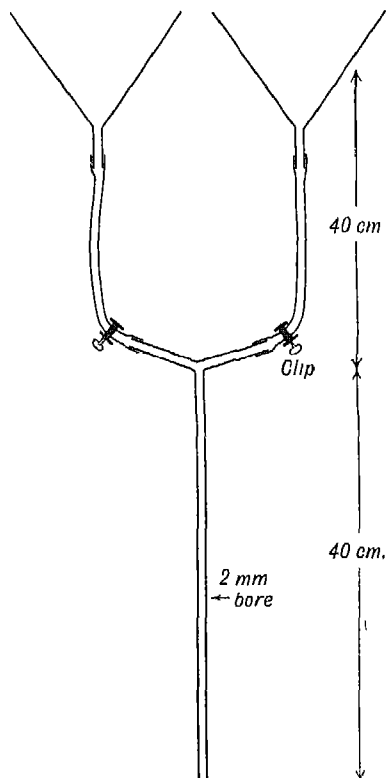


FIG. 48.

carbonic acid is reformed from the dissolved carbon dioxide, and before it leaves the tube the solution becomes colourless. The length of the pink colour in the tube can be altered by adjusting the clips on the rubber tubes; diminishing the rate of the alkali flow will shorten it.



The ordinary electrolytic dissociation constant of carbonic acid is  $\frac{(\text{H}^+)(\text{HCO}_3^-)}{(\text{H}_2\text{O}_3 + \text{CO}_2)} = 3 \times 10^{-7}$ . From a knowledge of the constant of the equilibrium  $\frac{(\text{CO}_2)}{(\text{H}_2\text{CO}_3)}$ , the true dissociation constant  $\frac{(\text{H}^+)(\text{HCO}_3^-)}{(\text{H}_2\text{CO}_3)} = 5 \times 10^{-4}$  can be obtained. Carbonic acid, which can be represented as hydroxyformic acid, is therefore really stronger than formic acid (dissociation constant  $2 \times 10^{-4}$ ), just as hydroxy-acetic acid (dissociation constant  $1.5 \times 10^{-4}$ ) is stronger than acetic acid (dissociation constant  $1.8 \times 10^{-5}$ ).

## 95. A DILATOMETRIC LAW OF MASS ACTION EXPERIMENT

*E. J. Leeming*

The rate of hydrolysis of acetal,  $\text{CH}_3\text{CH}(\text{OC}_2\text{H}_5)_2$  has been studied dilatometrically by Bronsted and Wynne-Jones (*T. Farad. Soc.*, **25**, 59, 1929). The increase of volume during the reaction is quite large, 100 c.c. of 0.05M acetal solution expands by 20 cu. mm. on hydrolysis. The reaction has several features that would make it especially suitable as a school experiment on the law of mass action, the chief perhaps being the elimination of a large number of titrations. It is catalysed by hydrogen ions, but there is no change in hydrogen ion concentration as the reaction proceeds, because the acetal and also the alcohol and acetaldehyde produced by the hydrolysis are all neutral. In this respect it differs from most of the other hydrolytic reactions, which are usually autocatalytic. It is catalysed by hydrogen ions alone, and there is practically no general acid catalysis. Also, it is not normally a reversible reaction. Hence, in dilute solution of a given hydrogen ion concentration, the reaction is strictly monomolecular without any kinetic

complications. It has, however, a large and positive primary salt effect which is linear up to about 0.2 molar salt solutions. Owing to the simplicity of the catalytic effect, Bronsted proposed that the reaction should be used as a fundamental method of measuring hydrogen ion concentrations.

The dilatometer shown in the illustration is a modified form of one described in a recent paper (Palomaa and Salonen, *Ber*, 1934, **67**, B, 424). It consists of a bulb of about 50 c.c. capacity, shaped like that of a pipette. A graduated capillary tube, 20 cm. long and about 0.9 mm. in diameter, is sealed to the top of it. The tube sealed to the bottom of the bulb is bent upwards parallel to the capillary and is expanded at the top to form a funnel; this tube has a tap about level with the top of the bulb.

The reaction has a convenient speed when the acid concentration is about  $N/400$  and, if the acetal concentration is 0.05 – 0.1M, the expansion is within the capacity of the capillary. Equal volumes of solutions twice these strengths are mixed at a measured instant and the mixture is poured through the funnel into the dilatometer; if the tube above the tap is kept full of liquid during the filling, no air bubbles will be enclosed. The tap can be closed before the bulb is completely filled, and then opened a little so that the position of the meniscus in the capillary can be adjusted to a point near the bottom of the scale. The times when the meniscus reaches different points on the scale are read. The apparatus should be immersed in a large vessel, e.g. a 2-litre beaker, of water at room temperature; the temperature will then remain constant for some hours.

In one experiment, 50 c.c. of a solution containing 1

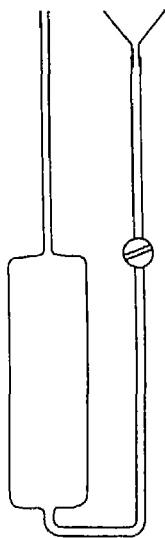


FIG. 49.

gm. of acetal, approx. 0.17M, were prepared and mixed with 50 c.c. of N/200 hydrochloric acid. The dilatometer was filled and the first reading of the level of the meniscus taken about two minutes after mixing the solutions. At first the meniscus rose 1 mm. in about twenty seconds, but forty minutes later the rate of rise had fallen to 1 mm. per minute. In 200 minutes the meniscus had risen 12 cm., and the reaction was about 90 per cent. complete. The final reading was taken after the apparatus had stood overnight, the temperature of the bath being the same as it was at the beginning. The total rise was 14.6 cm.

The initial reading,  $a$ , cannot be estimated directly, but can be found graphically. At any given time the amount of acetal that has not yet been hydrolysed, i.e.  $(a-x)$ , is proportional to the final reading minus the reading at that time. If the logarithms of these numbers are plotted against the times, the graph is a straight line, the equation of which is

$$t = \frac{1}{k} \{ \log a - \log (a - x) \}.$$

The velocity constant of the reaction can be obtained from the slope of the graph. Alternatively, the line can be extrapolated to cut the axis of time when  $t = 0$  and the intercept is  $\log a$ . The velocity constant may then be calculated from the readings at various times, using the value of  $a$  thus obtained. The graph of  $\log (a-x)$  against time is usually an excellent straight line for the first half of the expansion, after which the points often diverge and lie on a curve. This is probably due to the fact that errors in the initial time and in the final reading of the level of the meniscus are more serious when  $(a-x)$  is small.

For successful working of the experiment, it is absolutely essential that the capillary should be quite freed from grease, and a thorough cleaning with chromic acid mixture is necessary. Even with a clean capillary, tapping is

required to free the meniscus. The apparatus need not, however, be dried carefully after cleaning, because a little water will not alter the concentration of the acid appreciably. The tap in the right-hand tube should be specially ground to prevent leakage. The acetal should be free from acid and may be neutralized by solid sodium carbonate. The distilled water used in making up the acetal solution should be fresh.

The velocity constants at 20° C. that are given by Bronsted and Wynne-Jones for hydrochloric acid concentrations of 10, 8, 6, 5 and  $3 \times 10^{-4}$  normal are 19.2, 15.3, 11.8, 10.1 and  $6.55 \times 10^{-3}$  mins<sup>-1</sup> respectively. Palomaa and Salonen state that the temperature coefficient of the reaction  $K_{35^\circ}/K_{25^\circ}=3.5$ .

## COLLOIDS

### 96. COLLOIDAL SILVER (CAREY LEA)

#### *G Fowles*

A splendid silver sol is obtained by this method, but the preparation cannot be completed in one lesson. 5 gm. of ordinary yellow dextrin and 5 gm. of caustic soda are dissolved separately in water, the solutions mixed and diluted to 250 c.c.

Transfer the alkaline dextrin to a large flask and add slowly a solution of 3.5 gm. of silver nitrate in 20 c.c. of water, keeping the contents of the flask well agitated; silver oxide is precipitated. Allow the flask to stand for an hour—or until the next lesson or on a water-bath for twenty minutes. the brown silver oxide is reduced by the dextrin to black silver which is in the colloidal state. To some of this colloidal suspension add an equal volume of alcohol. The dextrin, caustic soda and oxidized products remain dissolved in the alcohol, but the silver is precipitated and settles in an hour or more. At this stage decant the liquid off; this is quite easily done since the silver, in a tar-like mass, adheres to the walls of the

vessel; wash with a little alcohol. On now adding a large volume, say 500 c.c., of water, the silver is peptized and a perfectly clear liquid—green by reflected light, brown-red by transmitted light—is obtained.

To some of this sol add dilute hydrochloric acid; no silver chloride is precipitated because no silver is in solution, but the acid coagulates the sol and a dense black precipitate of metallic silver is obtained. The sol responds in the same way when treated with a variety of other electrolytes.

#### 97. RELATIVE RATES OF DIFFUSION OF CRYSTALLOIDS AND COLLOIDS

*G. H. Locket*

The following extremely simple experiment is quite effective. A piece of tubing ( $\frac{3}{4}$  in. diam. and 2–2½ m. long) is filled with gelatine in which has been dissolved some potassium iodide. A little of the jelly is removed from each end of the tube and is replaced at one end by an iodine solution (in KI) and at the other by a starch solution, both of which are kept in place by corks. The tube is now left for one or perhaps two days, when the iodine will be seen to have diffused into the jelly, but the black ring where it meets the starch is only a few millimetres from the starch solution.

#### 98. RELATIVE RATES OF DIFFUSION OF CRYSTALLOIDS AND COLLOIDS

*G. Fowles*

##### *Colloids*

*Arsenic Sulphide Sol and Potassium Chromate.*—This is shown in a spectacular and convincing way by the jelly experiments of Wolfgang Ostwald and A. A. Noyes.

Following Ostwald, prepare a quantity of 5 per cent. gelatin solution and pour the warm liquid into several

pairs of test-tubes or boiling-tubes until they are half-filled, placing the same volume of gelatin in each of the tubes forming a pair. These gels should be prepared before the lesson. In the first pair fill up one tube with arsenic sulphide sol and the other with a solution of the crystalloid potassium chromate of a strength to match as nearly as possible the tint of the arsenic sol and allow the tubes to stand under observation for a day or two. In a quarter of an hour, the potassium chromate will have penetrated the gelatin to a measurable distance; in twenty-four hours a splendid diffusion effect extending from 1 to 2 in. into the gel is observed, and after two or three days the gel is of a perfectly homogeneous colour. Meanwhile, the colloidal arsenic solution has scarcely stained the surface of the jelly on which it rests.

*Cuprammonium Sulphate and Ferric Ferrocyanide* —In another pair of tubes use a solution of cuprammonium sulphate as the crystalloid and one of ferric ferrocyanide as the colloid. The copper solution is made by adding strong ammonia to a strong solution of copper sulphate until the precipitate has just dissolved. This solution should be subsequently diluted to match as nearly as possible the ferrocyanide sol.

The ferricferrocyanide sol is prepared by mixing, in equivalent proportions, dilute solutions of potassium ferrocyanide and ferric chloride. Weigh out 1.055 gm. of potassium ferrocyanide ( $K_4Fe(CN)_6 \cdot 3H_2O$ , *M.W.* = 422), dissolve it in 250 c.c. of water, thus making a solution 0.01M: add to it 250 c.c. of an equivalent solution of ferric chloride.

It is not advisable to use a weighed quantity of ferric chloride for the preparation of this solution. Ferric chloride as usually purchased is a deliquescent mass of indefinite hydration, consequently, the actual content of ferric chloride is doubtful and the sol when made is almost certain to contain an excess of one electrolyte. This excess diffuses into the jelly below in the actual experiment, not in quantity sufficient to spoil the experi-

ment, but enough to reveal the experimenter's lack of control over his quantities. The direct determination of a ferric salt by iodimetry is now a simple operation; a strong solution should be prepared and standardized. The required volume of this solution is diluted with water.

A stable blue sol forms. It is advisable to show that a portion runs unchanged through a filter while another fraction is readily flocculated when warmed with dilute acid.

*Cuprammonium Sulphate and Ferric Ferrocyanide* (2). (*Method of A. A. Noyes*).—Some thick sticks of agar-agar jelly are prepared before the lesson. Agar-agar and not gelatin must be used in these experiments because agar does not stick to glass while gelatin adheres most tenaciously. The agar powder should be soaked in water for twenty-four hours; it then swells prodigiously. Solution is obtained on boiling. Beware of bumping, and if an enamel vessel is available, it is advisable to use it. Any attempt to dissolve the dry powder in hot water without the previous soaking is futile; a lumpy mass results which will not form a sol. Prepare a quantity of 4 per cent. solution and mould some sticks by pouring the warm liquid into an open tube corked at one end. If the sticks are 1–2 in. in diameter, they will keep their upright position when placed in a gas-jar. As a mould the writer uses a discarded 100 c.c. measuring cylinder which has lost its base: it makes sticks of a most convenient size. When the solution has set the jelly is most easily pushed out. Half-fill one gas-jar with a sol of ferricferrocyanide prepared as above, and a second jar with the electrolyte cuprammonium sulphate; stand one of the jelly sticks in each. After a day remove the sticks and cut a section. The electrolyte will be found to have diffused into the jelly; the section will be entirely blue and homogeneous in appearance while the jelly in the ferricferrocyanide sol is unchanged.

*Congo Red and Ferric Thiocyanate*.—For another pair of solutions use congo red in water as the colloid and a

0.01 ferric thiocyanate as the electrolyte. The solution of ferric thiocyanate is made by mixing equal volumes of 0.02M KCNS and 0.0066M FeCl<sub>3</sub> and adding 2–3 c.c. of concentrated hydrochloric acid to prevent hydrolysis. These two solutions make an excellent colour match.

## 99. PROTECTIVE ACTION OF COLLOIDS

*G. Fowles*

Prepare a 0.1M. solution of mercuric chloride by dissolving 6.8 gm. of the salt in 250 c.c. of water, and a 0.2M. solution of potassium iodide by dissolving 8.3 gm. in 250 c.c. of water. Dilute equal volumes of these solutions with two or three times their volume of water and then mix the liquids. The yellow form of mercuric iodide (rhombic crystals) is momentarily precipitated and immediately passes into the stable crimson form (tetragonal). Dilute another fraction of each solution with its own volume of 2 per cent. gelatin and again mix equal volumes of the reagents. The yellow form appears immediately, but in the colloidal state the yellow colour persists for several days.

Prepare 0.1N solutions of AgNO<sub>3</sub> and K<sub>2</sub>CrO<sub>4</sub>. Mix equal volumes of these in a tall cylinder: red silver chromate is precipitated and soon settles at the bottom. Next dilute 50 c.c. of each solution with 50 c.c. of 2 per cent. gelatin. On mixing equal volumes of these solutions, a red suspension forms which runs unchanged through a filter.

## 100. SWELLING OF COLLOIDS

*G. Fowles*

*Gelatin.*—Weigh a piece of sheet gelatin—say 1 in. square—immerse it in a beaker of water and observe its appearance over a few days. When the swelling process seems to have ceased, remove the swollen gelatin, dry it



with blotting-paper and find the increase in weight. Let it stand in the air for a few days until the shrinking seems to have ceased; on weighing again, approximately the original weight is found. The agreement will not be exact unless the gelatin was free from water-soluble substances, the humidity of the air the same on both occasions, and the gel allowed to swell or shrink in air until equilibrium was reached.

*Agar*.—In a 100-c.c. measuring cylinder place powdered agar and press it well down with a rammer made by a cork fixed on the end of a rod, until the layer occupies a definite volume, say 5 c.c. Now pour in water (using 15 times the volume of the agar) and keep the system under observation. Swelling soon begins and subsequently the agar occupies 8–10 times its original volume.

*Crêpe Rubber*.—Cut pieces of a definite shape, say  $\frac{1}{2}$  in. squares, from a sheet of crêpe rubber. Place these pieces severally in corked flasks, sufficiently large to allow the rubber to swell without the shape being distorted, containing such rubber solvents as benzene, toluene, carbon tetrachloride, westron, westrosol, etc. In a few hours the rubber has swollen enormously, but for a long while the pieces retain their shape, the little squares becoming big squares. After a time the rubber forms a gel with the solvent.

## 101. DETERGENT ACTION OF SOAP

*G. Fowles*

Place 50 c.c. of water in a 100-c.c. stoppered cylinder and 50 c.c. of aqueous soap solution (0.25–1.0 per cent) in a second cylinder. The soap solution is made by adding the requisite amount of powdered soap to warm water. To each cylinder add a pinch of rouge or lamp-black and shake the liquids. These materials should be tested beforehand and the more suitable one chosen, if the material is too coarse the experiment is unsatisfactory.

The powder in the first cylinder eventually settles, leaving a perfectly clear liquid. While waiting for it to settle, pour some of it through a filter; the liquid comes through clear and colourless. Here again the experiment should be tried beforehand and a good quality quick-action filter paper selected; ordinary filter paper will probably allow some of the rouge or lamp-black to pass through.

The liquid in the second cylinder is a black or red suspension and will remain so. Pour some through a filter, using paper of the quality which has been found to retain the rouge; the red liquid runs through unchanged. As a variation, the soap solution may be poured into the filter cone containing the rouge after filtering the aqueous suspension; a red liquid passes through. This illustration of the detergent action of soap is modified from the original experiment of Spring.

Add dilute acid to the suspension of rouge, or a solution of alum or calcium chloride, indeed anything which will precipitate the stearate radical: the soap is decomposed and the rouge settles.

## 102. SAPONIN FOAM

*G. Fowles*

In a stoppered flask or cylinder shake up an aqueous solution of saponin (0.5–1 per cent.), an enormous volume of lather is produced which remains for hours.

Saponin is inexpensive, but if a supply is not at hand, grind up a horse chestnut and extract the powder with water. Filter the liquid and shake it; the lather formed is stable for hours.

## 103. FOAM AS A FIRE EXTINGUISHER

*G. Fowles*

The formation and application of foam is exhibited in the following way. A large volume, say 300–400 c.c., of

a cold saturated solution of sodium bicarbonate is prepared and saponin added to it till 1 per cent. of this substance is present. About 100 c.c. of a saturated solution of aluminium sulphate are also prepared. Alum will serve, but the former is far more effective. Place 25 c.c. of the carbonate solution in a 250-c.c. measuring cylinder and then about 5 c.c. of the aluminium sulphate; on mixing, a stiff foam is produced, which almost fills the cylinder. Obtain a thick-walled bottle, 300–500 c.c.; an ordinary flask is not suitable, for the pressure produced is liable to burst it. Close the bottle with a one-holed rubber stopper carrying a small right-angled tube to which a length of 3 or 4 in. of rubber tubing is attached. Pour 200–300 c.c. of the soda solution into the bottle; then stand in this solution 3 or 4 test-tubes filled with the aluminium sulphate solution. The tubes should contain a volume of solution equal to about  $\frac{1}{2}$  the carbonate solution. The liquids are mixed by inverting the bottle. A jet of foam is evolved which may be projected on to a piece of blazing wood previously soaked in petrol. The heavy carbon dioxide completely blankets the flames, and the colloidal aluminium hydroxide, carried up into the interface, assists by forming a series of non-combustible layers. The foam will persist for hours and prevent any re-ignition of the fire.

## ELECTROLYSIS

### 104. ELECTROLYSIS

*H. C. Palmer*

Two of the gas burettes (see No. 47, p. 106) are attached to a U-tube voltameter containing bench caustic soda electrolysed between platinum, nickel or silver electrodes. In series with the above, it is convenient to have several copper voltameters and one (or more) silver voltameter and an ammeter. The whole is then connected through

a lamp resistance with the mains. A current of half to one ampère may be employed.

This experiment is conducted by a form of boys, and if the ammeter is reliable, it is worth while to determine the average current and the duration of the experiment. The final calculations, shared by the whole form, provide a good review of the laws of electrolysis.

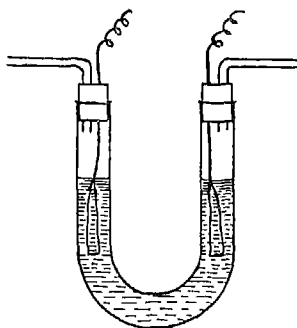


FIG 50.

## 105. CHEMICAL RECTIFIERS

*G. N. Pingriff*

Two kinds have been tried : the first had electrodes of aluminium and lead in a solution of sodium phosphate, though other electrolytes are said to serve just as well. The aluminium plate can act only as a cathode, the theory being that during that part of the cycle in which the aluminium should be the anode, a film of oxide forms and presents a barrier to the passage of the current. This cell rectifies a varying percentage of the current input, depending upon temperature and supply voltage. The lead is in the form of pipe through which cold water flows and the aluminium in the form of wide hollow cylinders (pieces of tube). The exact strength of the electrolyte is not important, but for complete rectification, the voltage should not exceed about 25 volts. On higher voltages, e.g. direct on 240-volt mains, there is sufficient excess of current in the forward direction to charge small accumulators. By using four cells suitably connected, it is possible to utilize both halves of the current cycle. Interesting details can be found in F. M. Perkin's *Practical Methods of Electrochemistry*.

## 106. CHEMICAL RECTIFIER FOR ELECTROLYSIS

*W. B. Barker*<sup>1</sup>

For rectification of alternating current from mains, the following apparatus has been thoroughly tested and found suitable.

Four glass or earthenware jars, holding approximately 1 quart of liquid each (battery jars or 7-lb jam-pots will do), are placed in line on a wooden base-board and filled with a saturated solution of *neutral ammonium*

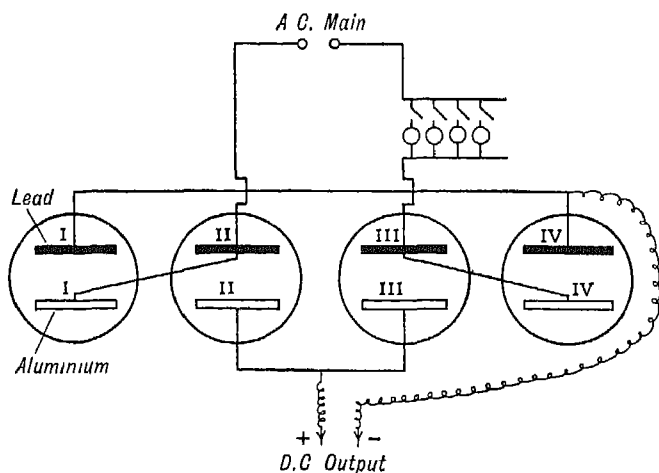


FIG 51.

phosphate to within 1 in. of the top. Sufficient heavy mineral oil (car lubricating oil) is poured on top of each jar to form a layer  $\frac{1}{2}$  to 1 in. thick. This entirely prevents "creeping" and evaporation of the solution and is essential. If this layer of oil is omitted, "creeping" becomes such a nuisance that the apparatus will need very frequent dismantling and cleaning up.

Suspended in each pot is a pair of metal plates, one of lead and the other of aluminium. These should be about

<sup>1</sup> For Valve Rectifiers see Series II, Part I, p. 250.

9 in. deep by 3 in. wide and not less than  $\frac{1}{8}$  in. thick. They can be fastened by screws to a narrow batten of wood, running horizontally above the pots and supported by uprights from the base-board at either end. Each plate should have two holes drilled at the top for fastening to the batten, the aluminium plates being fastened to one side and the lead plates to the other. While as much of the plates should be immersed in the pots as possible, they must not be allowed to touch the bottom of the pots or each other—otherwise “shorting” may occur. If the screws are provided with washers, they can be used as terminals.

For wiring connections it is best to use insulated single strand cable, and the wiring is carried out as follows (see Fig. 51):

	Pairs of Plates	Lead I Aluminium I	Lead II Alum. II	Lead III Alum. III	Lead IV Alum. IV
(a)	Connect	Lead II	to	Aluminium I	
(b)	„	„	III	„	IV
(c)	„	„	I	„	Lead IV
(d)	„	Aluminium II	to	Aluminium III	

Input from mains: one wire is connected to Lead II and the other to Lead III, *but* in one wire *must* be placed a suitable resistance (see below).

Output from rectifier: For positive wire tap centre of wire joining Aluminium II to Aluminium III.

For negative wire tap off from wire joining Lead I to Lead IV.

A suitable resistance for incorporation in the mains circuit can be constructed as follows. 4 32-c.p. carbon filament lamps of suitable voltage are mounted on a base-board by means of batten lamp-holders, and wired in parallel in one of the mains wires. If provided with switches in the holders, the amount of current passed can be regulated.

[N.B.—The more lamps put into circuit, the greater the amount of current passed.]

The total cost of the whole apparatus, including lamps

and the neutral ammonium phosphate ( $3\frac{1}{2}$ –4 lb.), should not exceed 25s. to 30s. For a current consumption of  $2\frac{1}{2}$  ampères, about  $1\frac{1}{2}$  ampères of rectified current are obtained, and this proves sufficient for all the usual experiments on electrolysis. When once constructed, no further attention is needed until the aluminium plates become so corroded that it is necessary to replace them. [Ours are three years old and show little signs of corrosion as yet !]

Two further points may be mentioned. (1) When the mains current is switched on, there may be a small interval before rectification is obtained. This is most noticeable when the apparatus is newly constructed or if brought into use after a long interval of rest. Usually this is only a matter of a few seconds. (2) Owing to evolution of gas from the plates some frothing of the oil occurs, but the froth rarely overflows and the little which may do so is easily wiped off the pots.

#### 107. TANTALUM RECTIFIER

*W. Bryan Chivers*

Many schools having A.C. supply require a simple, trouble-free rectifier for accumulator charging and D.C. experiments. The tantalum rectifier seems to be comparatively unknown, and yet it is a robust instrument which will stand considerable momentary overloads, is free from the messiness of the ordinary electrolytic rectifier, and compares favourably in efficiency with the "dry" rectifiers.

The diagram shows the details of construction. Tantalum is sold in thin foil strips and costs about 6s. per square inch. Each square inch of foil will handle up to 1.5 ampères at a peak voltage of 100 volts, so that the size and cost of the tantalum electrode for various requirements may be readily calculated. Since there is no wear of the electrode, the first cost is the only cost, and in the long run this rectifier is much cheaper to run

than other electrolytic types. The electrolyte is ordinary accumulator acid, S G. 1.25, to which a saturated solution of ferrous sulphate has been added in the proportion of 1 part of solution to 16 parts of acid. To prevent loss by evaporation, the cell should be enclosed (a large glass accumulator case acts admirably) and any evaporation loss must be made good with distilled water only. The lead electrode is good quality heavy sheet, carefully

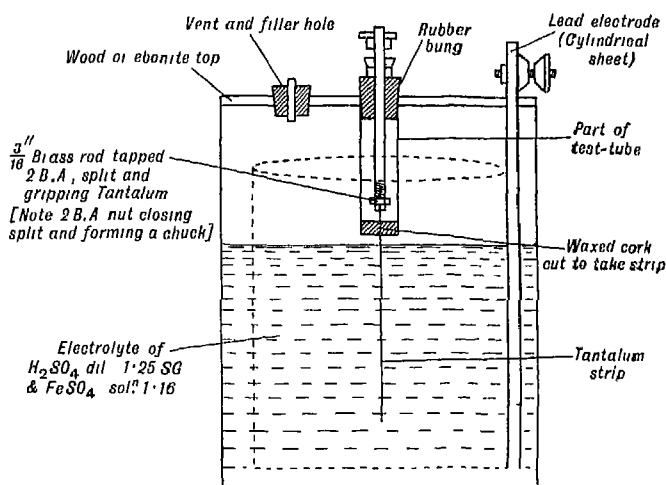


FIG. 52.

cleaned from grease and bent into a cylinder with the tantalum electrode as centre. Such a cell, with 3 pints of electrolyte and 1 square in. of tantalum immersed, will handle up to 1.5 ampères for considerable periods, but care must be taken that the temperature does not rise unduly.

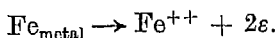
## 108 CORROSION OF IRON

*E. J. Bowen*

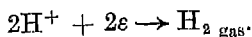
(1) When acids act on the more electropositive metals, e.g. zinc or iron, impurities behave as cathode areas and



the pure metal forms the anodic areas, and corrosion takes place by processes such as: (a) at the anodes,



The electrons liberated run through the body of the metal to the cathode areas, leading to the cathodic reaction (b) .



The metal is dissolved away at the anodes and hydrogen gas is liberated at the cathodes

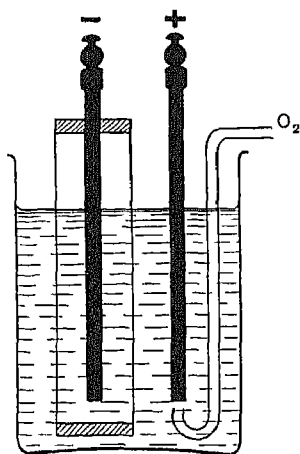
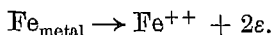


FIG. 53

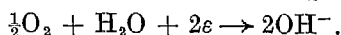
The normal action of moisture and air on iron to produce rust does not involve solutions sufficiently acid for the above process to take place, and the electrolytic action of corrosion is of the "oxygen depolarized type". The features of this are shown by the following experiment. An iron or steel rod, 6-8 in. long and 0.1-0.25 in. diameter, is cleaned very thoroughly with emery paper and cut into two electrodes of equal length. One electrode

is fixed through a cork into a very small porous pot, or into a cylinder made of parchment paper with cork ends. The pot is placed in a small beaker, in which is put the other electrode with a gas-delivery tube arranged below it (Fig. 53). A solution of sodium chloride is placed inside and outside the porous pot, and the electrodes are connected to a millivoltmeter (electrode in the porous pot to the - terminal.) When a stream of air or oxygen is blown over the outer electrode a large deflection is obtained, which slowly returns to near zero when the stream of gas is cut off. The interpretation of this result is that in presence of oxygen

iron becomes cathodic, i.e. more "noble," with respect to iron protected from oxygen. The oxygen, in fact, makes the iron passive, though to a lesser extent than other oxidizing agents, such as potassium dichromate or nitric acid are known to do. At the anode in the porous pot, i.e. the electrode protected from oxygen, iron dissolves.



The electrons run round the circuit to the cathode, where the depolarized cathodic action takes place.



If the cell is left short-circuited for a while, this mechanism can be verified by testing the liquid in the porous pot for ferrous ions with potassium ferricyanide and the outer liquid for hydroxyl ions with phenolphthalein.

(2) The important fact that iron corrodes where the oxygen has *least* access is simply demonstrated by hanging a cleaned sheet of iron vertically and almost immersed in a beaker of dilute salt solution. Rust forms on the bottom half of the sheet while the top half is hardly affected.

(3) Another striking experiment is the use of the "ferroxyl" indicator. A sheet of iron is cleaned carefully with emery and several large drops, 1-1.5 cm. across, of a sodium chloride solution to which phenolphthalein and a little potassium ferricyanide have been added is placed upon it. Almost immediately the circumference of the drop goes pink, showing the production of  $\text{OH}^-$  ions at cathodic areas where the oxygen has free access, while after a time the production of ferrous ions at the centre of the drop will be shown by the appearance of a blue precipitate. Where the anode and cathode liquors meet, a colloidal precipitate of ferrous hydroxide, oxidizing in air to ferric hydroxide, can be observed. This is rust, a purely secondary product in the corrosion of iron, which begins as ferrous hydroxide formed as described, and becomes more or less changed

by the absorption of oxygen and carbon dioxide. In the above experiments, salt solutions are used because of their high electrical conductivity; when iron rusts under natural conditions, the conductivity of the water condensed on the surface is due to carbon dioxide or other acid substances, sodium chloride if near the sea, etc

### LE CHATELIER'S PRINCIPLE

#### 109. LE CHATELIER'S PRINCIPLE

*G. Van Praagh*

Le Chateher's principle may be very simply demonstrated in the school laboratory by the reaction  $\text{N}_2\text{O}_4 \rightleftharpoons 2\text{NO}_2$ . The forward reaction occurs, at constant volume, with an increase in pressure and a deepening of colour, so if the pressure of a mixture in equilibrium is reduced, according to the principle, the equilibrium moves to the right, the change being made evident by a deepening of the colour.

Nitrogen dioxide is passed at room temperature into a vessel fitted with two taps, until the colour is pale orange. The taps are then closed. One is connected to the water pump and opened momentarily. The colour becomes pale yellow, owing to removal of gas by the pump, but within a second or so, it returns almost to its original depth, owing to an increase in concentration of the  $\text{NO}_2$  caused by further dissociation following the reduction in pressure.

### MOLECULAR WEIGHT

#### 110. SIMPLIFIED VICTOR MEYER APPARATUS

*H. C. Palmer*

The apparatus (Fig. 54) has been in use at Oundle for some years. The results obtained with it have been uniformly good. It has proved to be far less vulnerable

than the traditional apparatus. Owing to the simplicity of its construction and ease of assembly, it can be used with success by forms of boys working individually or in pairs.

The smaller stopper, E, should fit into a tube of approxi-

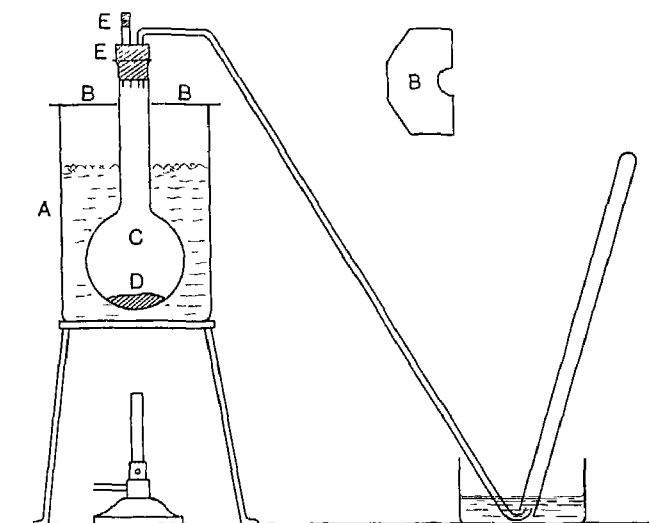


FIG 54

- A. Heavy gauge tin canister.
- B. Tin half-hds, cut to fit the neck of the flask.
- C. Kjeldahl flask.
- D. Well dried sand.
- E I.R stoppers.

mately  $\frac{1}{4}$ -in. internal diameter. The small displacement of air on inserting this stopper is then negligible

The quantity of volatile substance introduced should be such as to displace less than 50 c.c. of air into the measuring tube.

The apparatus rapidly comes to equilibrium, both before and after the volatile body is introduced. A good depth of water in the canister is advisable.

# 111. SIMPLE MODIFICATION OF VICTOR MEYER'S METHOD FOR SUBSTANCES OF HIGH BOILING POINTS

*H. C. Palmer*

B (Fig 55) is a long, wide test-tube, of such dimensions that the liquid F condenses about two-thirds of the way

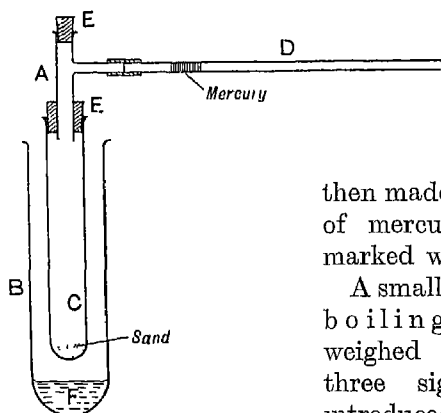


FIG. 55.

up. The jointed tube, A, is fitted with rubber stoppers, E, and flexibly joined to the tube, D, which is

then made level. An indicator of mercury has its position marked with gummed paper.

A small quantity of the high boiling-point substance, weighed to an accuracy of three significant figures, is introduced—usually in a capillary quill tube. The displacement

of the mercury indicator is measured and the volume of gas deduced, preferably by weighing that amount of mercury which fills the length of the tube, D, over which the displacement takes place. F is a high boiling-point liquid

# 112. MODIFICATION OF RICHARDS'S METHOD FOR FINDING FREEZING POINTS OF SOLUTIONS

*H. C. Palmer*

Instead of a flask, as shown in Fig 211 (*Science Masters' Book*, Series I, part 2, p. 86), a *thermos* flask is used. The method of working is otherwise identical. Excellent results are obtainable with solutions of all the easily titratable reagents.

113. MOLECULAR WEIGHTS BY THE  
FREEZING-POINT METHOD*H C Palmer*

Instead of the customary method of having an air-jacketed tube, it is quite satisfactory to use test-tubes with very thick walls. These have a similar effect and, in addition, have the advantage of strength

114. LABORATORY EXERCISE ON THE  
ASSOCIATION OF PHENOL*F. A. Philbrick*

It is well known that partition experiments between benzene or toluene and water will show benzoic acid to be entirely associated into double molecules in the non-aqueous layer. By using phenol instead of benzoic acid, it is possible to make a quantitative study of the equilibrium between single and associated molecules. Chlorobenzene is used as solvent

In the water layer, freezing-point measurements have shown that the phenol (concentration  $W$ ) is wholly monomolecular, while at the concentrations studied ionization is negligible. In the chlorobenzene layer, the phenol (concentration  $L$ ) is in single and double molecules in equilibrium (concentrations  $A$  and  $B$  respectively, whence  $A + 2B = L$ ). The association-constant  $K$  is  $B/A^2$ . Then the observed partition ratio  $P$  is

$$\frac{L}{W} = \frac{A}{W} + \frac{2B}{W} = P_0 + 2KP_0^2W.$$

Accordingly, if  $P$  is plotted against  $W$ , a straight line is obtained from which  $P_0$  and  $K$  can be calculated. If the concentrations are in gram-molecules per litre,  $P_0$  is 1.44 (at 25°) and  $K$  is 0.65. Up to at least 0.13 M in the

chlorobenzene, the experimental results are in close agreement with theory.

Proceed as follows : prepare an approximately 0.17 M solution of phenol in water and standardize it by the bromate-iodide method. In a stoppered vessel mix 100 c.c. of this solution with a known volume (or weight) of chlorobenzene, taking about 70 c.c. Shake well, allow to settle, and withdraw portions of the (upper) aqueous layer for analysis. Repeat with more dilute aqueous solutions until a series of points connecting P and W has been obtained. In all experiments the volume ratio should be about 10 of water to 7 of chlorobenzene.

The bromate-iodide method of analysis described in the standard textbooks, such as *Kolthoff*, gives exact results in experienced hands, but occlusion of bromine and adsorption of starch-iodide may cause difficulty. This can be avoided by adding, immediately before the titration with thiosulphate, sufficient carbon tetrachloride (very little is required) to dissolve the precipitate of tribromophenol. The titration is then carried out in a stoppered vessel, with constant shaking, but without the use of starch. The end-point is ideal.

Further discussion can be found in the *J. Amer. Chem. Soc.*, **56**, 2581 (1934).

## 115 SIMPLE MODIFICATION OF THE COTTRELL APPARATUS

*H. C. Palmer*

The "pump tube," A (Fig. 56), is about 4 in. high and is constructed at its upper end so as to hold the thermometer concentrically. The diameter at B is about  $\frac{1}{4}$  in. greater than the diameter of the thermometer bulb. This apparatus gives very steady readings and excellent results with a narrow thermometer, graduated in tenths of degrees.

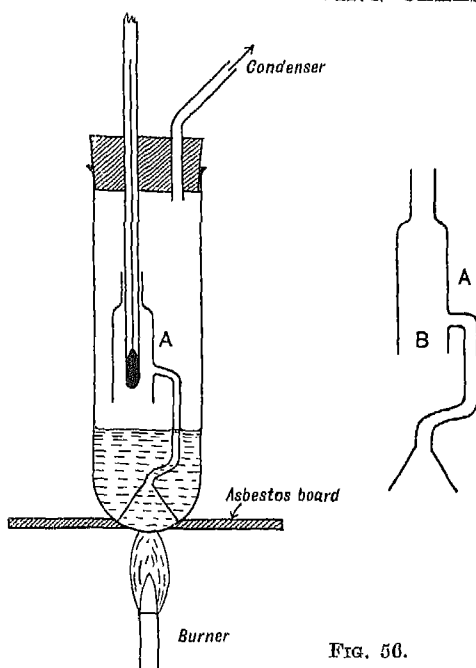


FIG. 56.

## OSMOSIS

## 116 OSMOSIS THROUGH LIVING CELLS

*G. Pallister*

Peel two large potatoes and cut one end off each, so as to obtain a flat surface. Hollow out the insides,

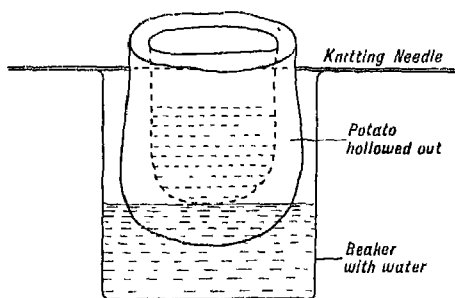


FIG. 57.



leaving fairly thick walls. Boil one of the potatoes for a few minutes, to kill the cells, and place about half a tea-spoonful of salt or cane sugar inside each potato. By means of a knitting-needle stuck through the top, suspend each potato in a beaker of water, the lower part only being immersed. After a few hours, the living potato will be seen to be nearly full of liquid, while the boiled one will not have changed.

### 117. OSMOSIS IN LIVING CELLS AND CHANGE IN WEIGHT

*G. Pallister*

By means of a cork borer, obtain cylinders of potato tissue of about a centimetre diameter, and cut these into discs of approximately 2 mm. thickness. Pick out several sets of ten, lightly dry them on blotting-paper and weigh

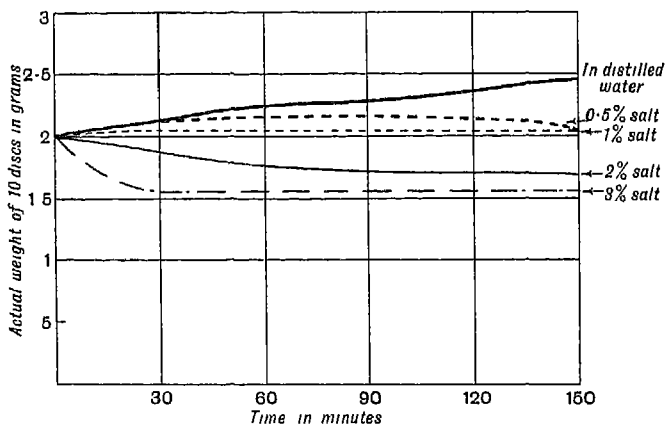


FIG. 58.

on watch-glasses. Place one set in distilled water, another in 1 per cent. salt solution, another in 2 per cent. salt, and so on. After half an hour, take them all out, dry as before and weigh again. Repeat every half-hour,

and draw graphs to show the percentage change in weight as time goes on.

The method can also be used to ascertain the suction pressure of the cells, which is equal to the osmotic pressure produced by that concentration of salt solution which causes no change in weight of the tissue.

RECORD OF EXPERIMENT BY SIXTH-FORM BOYS

	Initial Weight gm	Weight after 30 mins gm	Weight after 90 mins gm	Weight after 150 mins gm
In distilled water	1.97	2.11	2.28	2.44
0.5 per cent. salt	2.02	2.12	2.17	2.07
1.0 per cent. "	1.99	2.06	2.05	2.04
2.0 per cent. "	1.99	1.87	1.71	1.64
3.0 per cent. "	1.99	1.57	1.56	1.54

## 118. OSMOSIS WITH VISIBLE MOVEMENT

*E. T. Harris*

This apparatus shows in a way that is easily visible the osmotic movement of liquids and the consequent hydrostatic pressure. It is not quantitative, the membrane employed being partly permeable to the solute.

The membrane is Cellophane (sheet cellulose), the thinnest kind, sold as No 300, Standard Type, thickness 0.00081 in. A piece of this, 3 or 4 in. square, is folded neatly over one end of an open glass tube, about 3 in. by 1 in. wide, the edge of the tube having been rounded in the flame. It is then bound round evenly and tightly with about six turns of thin string, and tied. The string and cellophane are then made wet, when the shrinking of the string and the softening of the membrane produce a watertight joint, which may be tested by placing water first in the tube.

The tube is then filled nearly to the top with (to choose a familiar solution with a high osmotic pressure) golden syrup. In order that the liquid may show up well in the narrow tube, ink or other coloured liquid is poured on top of the syrup. A rubber stopper is inserted, carrying a

long piece of thermometer tubing of  $\frac{1}{2}$  mm. to 1 mm. bore. Since the ink frequently rises right up the narrow tube on inserting the stopper, the stopper is provided also with a narrow side-tube equipped with rubber tubing and a clip.

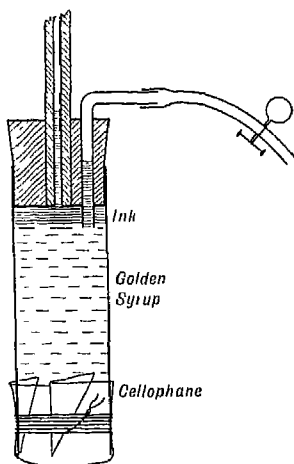


FIG. 59.

starting-place, or to bring it back again whenever it threatens to run over the top.

It is sometimes convenient to replace the thermometer tubing by "quill" tubing of about 3 mm. diameter and to omit the side-tube. The movement is then slower, but less attention is required because of overflowing, and the apparatus is more easily fitted up by pupils. The wide tube can be held in a clamp over a beaker of water and lowered into it when required. After a slight initial fall, the liquid in the tube will

be seen to rise quite rapidly. The motion is made more obvious by placing on the narrow tube small rubber rings cut from rubber tubing.

Brine or alcohol may be substituted for the syrup. They are easier to pour in, but salt and alcohol leak through the membrane more quickly than sugar.

## 119. COLLODION SACK FOR OSMOTIC PRESSURE DEMONSTRATION

*F. Briers*

Fill a perfectly dry boiling-tube with a moderately thick solution of collodion in ether, and allow to stand, constantly breaking the skin which forms at the top of the tube, until all air bubbles rise. Carefully pour out

the ether collodion, invert the tube and drain for a short time. Dry out for a very short time with a current of cold air, and remove the skin around the edges. Fill with cold *distilled* water and immerse tube completely in a jar also filled with cold distilled water, when the collodion sack can easily be removed and kept under cold distilled water until required. If necessary, the sack can be stretched slightly to secure a good fit over a rubber bung, through which passes two tubes, one acting as the pressure indicator. The sack is held firmly around the bung by binding into position with several turns of stout thread. Sugar solution, etc., is introduced into the sack *via* the pressure indicator and the second short tube then sealed off. Simply immerse sack completely in cold distilled water, when, after a short time, solution rises in the indicator tube.

## 120. DIFFUSION OF GASES THROUGH A WET MEMBRANE

*W. L. Francis*

A piece of parchment membrane, which has been well soaked in water, is sealed over the end of a wide glass tube which is full of air and connected with a manometer. The moistened membrane is then surrounded, first by an atmosphere of hydrogen, and second, by an atmosphere of carbon dioxide or ammonia. The movement of the manometer liquid shows that penetration through the damp membrane is far faster for ammonia and carbon dioxide, which are soluble in water, than for hydrogen, which is far less soluble. With a dry membrane, the relative speeds of diffusion, being governed by the densities and not the solubilities, would be in the reverse order.

## 121. CASTOR OIL AS A SEMIPERMEABLE MEMBRANE

*W. L. Francis*

Layers of water, castor oil and methyl alcohol are placed in that order in a test-tube, care being taken to avoid mixing. The layer of castor oil should be as thin as possible. The tube is corked to prevent evaporation and left undisturbed. In the course of time, the methyl alcohol layer diminishes in depth and the water layer increases. Finally, the presence of methyl alcohol can be detected in the aqueous layer by withdrawing a sample and treating with salicylic acid and sulphuric acid. The odour of methyl-salicylate (oil of wintergreen) is noticed. The methyl alcohol has dissolved in the castor oil, diffused through to the water-oil surface and distributed itself between the oil and water phases. The oil is impermeable to water because it cannot dissolve water. The methyl alcohol penetrates because it is (1) soluble in the oil and (2) also soluble in water.

## 122. PHENOL-WATER SEMIPERMEABLE MEMBRANE

*W. L. Francis*

Water and phenol are shaken together until two mutually saturated layers are formed. Part of the more aqueous solution is now saturated with calcium nitrate and placed at the bottom of a test-tube. On this is carefully pipetted a thin layer (less than 1 cm) of the phenol saturated with water, and finally, on this a layer of the water saturated with phenol. In the course of a few days, the bottom layer increases at the expense of the top layer and the middle layer rises up the tube. The phenolic layer is behaving like a semipermeable membrane. The calcium nitrate solution has a high osmotic pressure compared with the aqueous solution of phenol. The inter-

vening layer of phenol is impermeable to calcium nitrate but permeable to water, which therefore diffuses steadily into the solution of higher osmotic pressure. In this illustration, the motive power of the diffusion is the difference of osmotic pressures of the aqueous layers, but the mechanism of osmosis is the solution of the water in the intervening non-aqueous layer

### PARACHOR

#### 123. DETERMINATION OF A PARACHOR

*E. J. Williams*

The density of a pure liquid, and the surface tension relative to water, are determined as nearly as possible at the same temperature, so the use of a thermostat is desirable, though not essential if the determinations are made within a short period of time in a room where the temperature is uniform over the period.

The surface tension, relative to water, is determined by means of Traube's Stalagmometer.

The apparatus (Fig. 60) consists of a capillary tube, with a flattened, polished end, joined on to a wider tube with a bulb, as sketched. Marks are made above and below the bulb, but it is not necessary to know the volume between these. The apparatus is fitted into a bottle by means of a rubber stopper as illustrated, a long tube also being fitted so as to maintain atmospheric pressure when the bottle is immersed in a thermostat

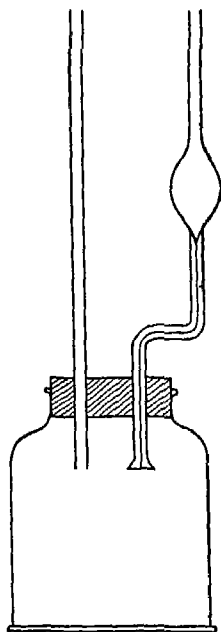


FIG. 60

The stalagmometer tube is  
SMR—112

thoroughly cleaned with chromic acid and dried by aspirating through it a current of warm air, rendered dust-free by filtration through cotton-wool. Water is drawn in to fill it above the upper graduation, and this is allowed to drop out, the number of drops falling between the upper and lower graduations being counted. This is repeated to obtain agreement, and if one always works at the same temperature, the instrument is calibrated for water.

The stalagmometer is dried as before, filled with the liquid under examination, and the number of drops again determined.

(The original article in the *School Science Review* gives the results obtained with allyl alcohol, nitro benzene and ethyl cinnamate.)

If  $\gamma_1$  = surface tension of liquid,  
 $\gamma_2$  = surface tension of water,  
 $d$  = density of liquid,  
 $n_1$  = number of drops of liquid,  
 $n_2$  = number of drops of water,

$$\gamma_1 = \frac{\gamma_2 n_2 d}{n_1}$$

and, if  $M$  = molecular weight of liquid,

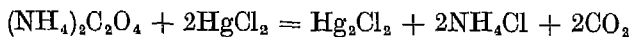
$$[P] = \frac{M}{d}(\gamma_1)^{\frac{1}{4}}.$$

## PHOTO-CHEMISTRY

### 124. PHOTO-CHEMICAL REACTIONS

#### *A. Pickles*

*The Reaction between Ammonium Oxalate and Mercuric Chloride.*—The reaction represented by the equation ·



is very slow in the dark, especially if the solutions are dilute. In sunlight, or on exposure to the light from a powerful electric light, deposition of mercurous chloride

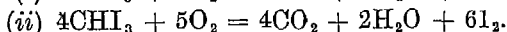
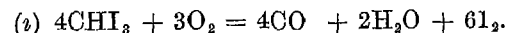
occurs. This fact has been known for a long time and has, indeed, been suggested for application in actinometers.

There are several points to consider, however, in the experimental investigation. If artificial light is used, say a 500-watt lamp, heating effects are considerable, and the temperature of the reactants rises rapidly if the lamp is too near their surface. There is also a marked period of induction before mercurous chloride is noticed, possibly due to the solution having to reach saturation-point, though this should not take so long as it does in practice since the solubility of mercurous chloride is only 0.0002 gm. per litre at 18° C. The reaction is also retarded by dissolved oxygen in the water from which the solution is made, so that freshly boiled distilled water is advisable.

The following results were obtained using a 500-watt focuslite lamp on deci-normal solutions, the reaction being followed by means of N/10 permanganate, after the mercurous chloride had been filtered off. During the experiment, the temperature of the reacting solutions rose from 17° C. to 28° C. in spite of the containing vessel being surrounded by cold water. The lamp was about 10 in. above the surface of the liquid, which was continuously stirred.

Time (mins.).	Amount N/10 Permanganate for 20 c.c. Solution	Time (mins.)	Amount N/10 Permanganate for 20 c.c. Solution
0 . . . . .	0.0	12	8.9
9 . . . . .	9.10	17	8.5

*Photo-Chemical Decomposition of Iodoform.*—The reaction that occurs may be represented by the equations.



A suitable concentration is N/40 solution, and carbon tetrachloride the best solvent to use. Alcohol is not suitable since the decomposition in this medium is very slow, evaporation occurs, and the solution is inflammable. Benzene may be used, but since it is inflammable, it



does not lend itself to ordinary laboratory work as does carbon tetrachloride.

The following results were obtained using N/40 solutions of iodoform in carbon tetrachloride.

(a) Focuslite lamp, 500 watts. Distance about a foot from solution surface. Temperature, from 20° C. to 27° C.

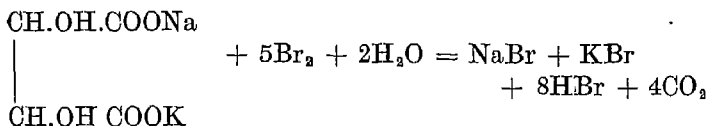
Time (mins.)	Vol. N/40 Thiosulphate for 10 c.c. of Solution	Time (mins.)	Vol. N/40 Thiosulphate for 10 c.c. of Solution
2	1 05	8	8 10
5	5 10	12	10 0

(b) 100-watt, gas-filled lamp. Plain glass Temperature from 20° C. to 22° C. Distance, 1 ft from liquid surface.

Time (mins.)	Vol. N/40 Thiosulphate for 10 c.c. of Solution	Time (mins.)	Vol. N/10 Thiosulphate for 10 c.c. of Solution
19	1 20	57	4 10
37	2 50	90	6 80

In both experiments it will be found that decomposition of iodoform will proceed to some extent after withdrawal of the light unless, of course, the iodoform has been fully decomposed as in results (a) above.

*Reaction between Bromine and Rochelle Salt.*—The oxidation represented by the equation :



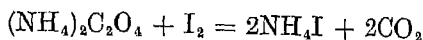
proceeds slowly in the dark, but is considerably accelerated by exposure to daylight, or to the light of a 100-watt lamp.

The concentrations of solutions used were, Rochelle salt, 10 gm. per litre, bromine water, N/25; sodium acetate, which is added to prevent increase in concentration of hydrogen ions, 14 gm. per litre; the disappear-

ance of the bromine was followed by using potassium iodide and N/25 thiosulphate.

Time (mins.)	Vol. Solution c.c.	Vol. N/25 Thiosulphate c.c.
5	20	19.0
10	20	10.2
20	20	5.4
30	20	3.80
40	20	2.90

*Reaction between Iodine and Ammonium Oxalate.*—The reaction is an oxidation and may be represented by the equation below, which does not show possible intermediate reactions.



The concentration of the respective solutions was ; iodine N/100, ammonium oxalate 18 gm. per litre.

In the experiment, 20 c.c. of ammonium oxalate solution were added to 100 c.c. of iodine. 10 c.c. of the mixture were withdrawn at definite intervals and the amount of iodine estimated by N/100 thiosulphate. Day-light was used (May, bright light, but not in actual sunshine).

Time (mins.)	Volume of N/100 Thiosulphate c.c.	Time (mins.)	Volume of N/100 Thiosulphate c.c.
10 .	3.75	50	2.30
20 .	3.25	75	1.80
30 .	2.95	100	1.60
40 .	2.60		

## SOLUBILITY

### 125 ACCURATE DETERMINATION OF SOLUBILITIES AND PHASE EQUILIBRIA

*F. Briners*

The exact determination of a solubility or of a phase equilibrium, and their variation over a wide temperature range are not easy, especially at fairly high temperatures. The greatest difficulty lies in obtaining a correct sample of the equilibrium solution for analysis, a difficulty parti-

cularly apparent at higher temperatures, when cooling of the solution is very rapid, with consequent ready crystallization. Also, in the case of a phase equilibrium involving, say, a three-component system, for instance, potassium sulphate, potassium nitrate and water, evaporation of the solution at higher temperatures must be prevented, if rapid displacement of the equilibrium is

not to occur. These difficulties can be overcome by use of the device shown in the diagram.

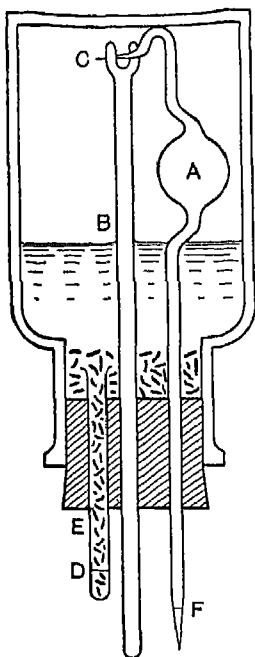


FIG. 61.

A bottle of 300–500 c.c. capacity (Fig. 61), charged with the complex (solid or solids plus water), is sealed with a tightly fitting rubber bung, which can be wired securely into position, a precaution obviously necessary at temperatures between 30°–100° C. Through this bung there are inserted an evacuated bulb A of about 20 c.c. capacity, a two-pronged fork B made from glass rod, and a quill tube E, rimmed out at one end and closed at the other. The end C of the bulb A (easily made by an amateur glass blower) is drawn out to a fine capillary, on which a glass-knife scratch is made, the prongs of B being so arranged that depres-

sion of B will cause the tip at C to break off, thus allowing a solution at any given temperature to be sucked into the bulb. The complex is brought to equilibrium by immersing the bottle completely in a thermostat and shaking for several hours, for instance, by strapping to a horizontal stirring shaft. At the end of this time the solid phase is allowed to settle on the bottom of the bottle, and the top of the bung is then

brought just to the surface of the liquid in the thermostat, the capillary tip C being broken off. The capillary is made sufficiently fine to prevent any solid phase from entering A. As soon as solution is seen at F, the bottle is immediately inverted under the surface of the liquid and then taken out of the bath, being allowed to cool down to atmospheric temperature in an inverted position. By removing the rubber bung, the bottle being held in an inverted position, liquor runs out of the bottle, after which the sampling bulb can be extracted with safety. The bulb is removed from the bung, the outside cleaned and dried, a knife-mark made at F, and the whole weighed. The other end of the bulb is then broken off and the contents washed out thoroughly with distilled water, made up to a known bulk and analysed. The dried pieces of the bulb are finally weighed, and from the results of the analysis the content of solid or solids per 100 gm. of solution can readily be calculated.

Owing to the possibility of pronounced solubility at higher temperature, e.g.  $90^{\circ}\text{C}.$ , it is necessary in some determinations to make other provision for removing A from the bottle on completion of an experiment. Often on cooling down from such high temperatures to room temperature, an almost solid crystalline mass is formed in the bottle, rendering removal of A most difficult. However, if the tube E is broken at D, a fine stream of fairly hot water can be injected into the crystalline mass, and by careful manipulation sufficient water can be introduced into the bottle either to dissolve, or to dislodge, the crystalline mass, thereby making the removal of the sampling bulb a comparatively easy matter.

This method of determining solubility is easy to carry out and is susceptible of great accuracy.

## THERMOCHEMISTRY

## 126 THERMOCHEMISTRY

*H. C. Palmer*

The cheap thermos flask is admirable for performing many of the experiments of thermochemistry. The water equivalent of the half-pint size—the most suitable for chemical work—is about 12 gm. Sufficient accuracy is attainable when mixing solutions by having one reagent present in the flask at known temperature, and then decanting the second solution over the bulb of the thermometer slowly into the flask. Finally, the flask is corked and well shaken, in order to render the temperature of the whole of the inner wall constant. Cooling corrections are not necessary, but the specific heat of the resulting mixture, unless this is very dilute, may not safely be assumed to be equal to that of water.

## EXPERIMENTS SUITABLE FOR RECEPTIONS, SPEECH DAYS AND GENERAL OCCASIONS

## 127. SIZE-WEIGHT ILLUSION

For this experiment, three hollow cylinders are required: A, about 3 in. in diameter and  $1\frac{1}{2}$  in. high. A convenient weight for this cylinder is 50–60 gm. B, a cylinder, 1 in. in diameter and  $1\frac{1}{2}$  in. high. This cylinder is weighted until it balances cylinder A. The third cylinder, C, has the same dimensions as B, but its weight is exactly half that of A or B. Any joints that show after the closing of the cylinders should be obscured with a coat of paint.

Cylinders A and C are placed on a bench; the visitor is asked to lift each *in turn* and decide which is the heavier. The only truthful answer is that C seems to be the heavier. The person, however, who is not going to be caught, will probably say that the larger cylinder

is heavier, in that case, substitute B for C. There can then be no quibbling about the decision. The visitor is then shown, by balancing on a pair of scales, that C is only half the weight of A, or, as the case may be, that B and A weigh the same. The most amusing part, however, is that after being shown that A and B weigh the same, people still maintain that B is heavier.

The explanation is that we are accustomed to gauge the effort necessary to do a piece of work and to put forth just that amount of effort and no more. The large cylinder comes away from the bench more easily than expected; on the other hand, the smaller cylinder drags a little and therefore feels the heavier. The visitor must not, of course, be allowed to take one in each hand until a trial has been made in the prescribed way.

## 128. TO ILLUSTRATE THE "NORMAL ERROR LAW"

G. W. Brewster

*Galton's Quincunx.*—When measurements are taken of the same characteristic for a large number of individuals—for example, the heights of boys of a definite age—there is a certain mean value of the measurements which occurs most frequently. Measures differing but little from the mean are somewhat less frequent; those differing more, less frequent still. The frequency of all the measures may be indicated graphically. In many cases, the law is that measures differing by  $x$  from the mean occur with frequency proportional to  $e^{-hx^2}$  where  $h$  is a constant. To illustrate the law, the biologist Galton devised the "Quincunx" (Fig. 62). It consists of a board, on which is an equilateral-triangle pattern of pins. The board is placed at about  $30^\circ$  inclination and shot are poured into a funnel at the top. In running down, the shot get deviated by striking the pins; they are received in a series of about 20 compartments, covered by a sheet of

glass (shown as a dotted rectangle in the figure), at a distance from the board a little greater than the diameter of the shot. The shot form piles in the compartments; the centre pile is highest, and those on either side fall off in height according to the law given above.

It will be found that a pattern of about 30 lines of pins,

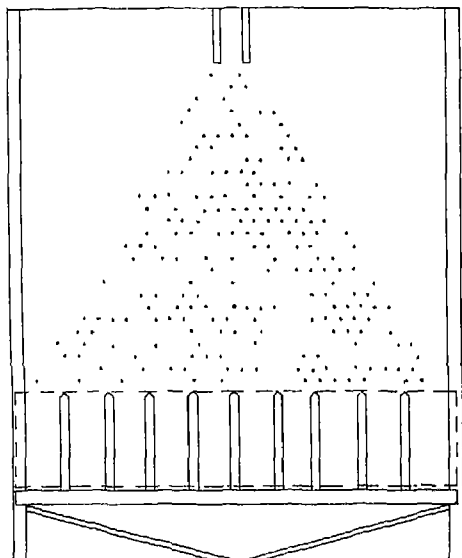


FIG 62.

forming equilateral triangles of 1 cm. side, gives good results with fairly large shot. It is convenient to have a movable base to the compartments, consisting of a bar which can be slid out, with a trough under it to catch the shot.

(This experiment is described in Whittaker and Robinson's *Calculus of Observations*, from which the idea has been taken.)

*A Penny-Tossing Experiment* (G. W. Brewster).—If  $n$  pennies are tossed a large number of times, there may be 0, 1, 2, 3 . . . . . ( $n - 1$ ),  $n$ , heads, and the

relative frequencies of these results are proportional to

$$1, n, \frac{n(n-1)}{2!}, \frac{n(n-1)(n-2)}{3!}, \dots, n, 1,$$

that is, the binomial coefficients in  $(a + b)^n$ .

If  $n$  is fairly large, it is known that a graph of these numbers against the numbers 0, 1, 2, . . . will give a curve of the normal frequency type. With 10 pennies

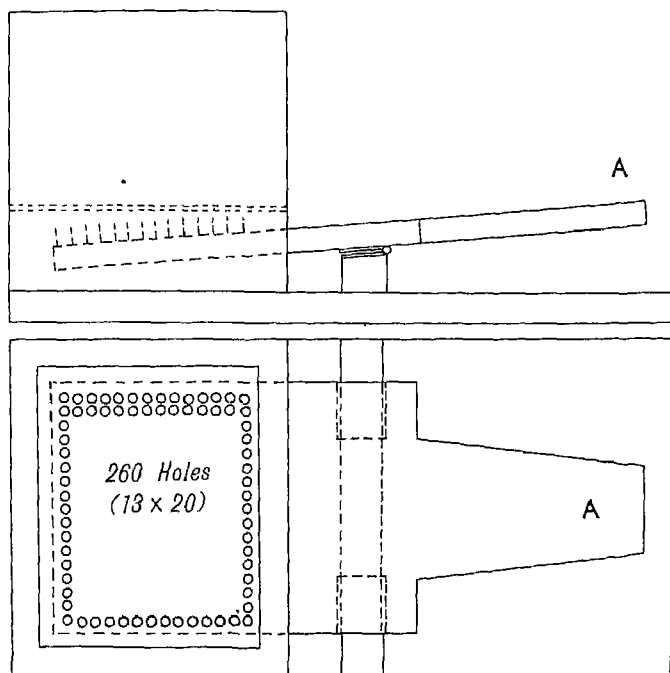


FIG. 63.

we should get in 1,024 trials, 1 result with 0 head, 10 with 1 head, 45 with 2, 120 with 3, and so on.

A simple apparatus (Fig. 63) to toss all 10 pennies at once (without having to deal with each coin individually) can be made as follows. A cubical box (about 6 in. side) has a string net at the top to prevent the coins jumping



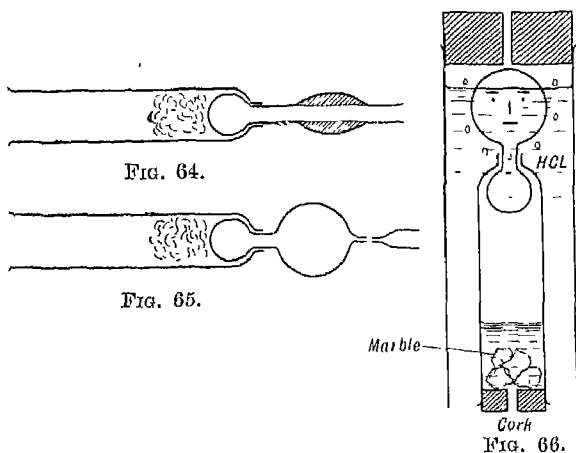
out, and a wooden base pierced with large holes, about  $\frac{1}{2}$  in. apart, preferably not regularly spaced. Under the box is a hinged board in which there are pegs so placed that they rise through the holes when the end is depressed. (These pegs may be 1 in. nails.) The whole is mounted on a base-board.

If 10 coins are put in the box, they will be thrown up when a blow is given to A, and the number of heads can be counted. It is convenient to put white paint on one side of the coins to facilitate counting the heads. By this apparatus, about a dozen trials can easily be made per minute. The results, as a rule, agree well with prediction and thereby satisfy the assumption that the tossing is really "at random."

## 129. DANCING MEN

*G. H. Locket*

Blow a small bulb in the end of a piece of glass tubing; also blow a hole in the bottom of a test-tube and wedge



the tube in (Fig. 64) with glass wool. Thicken the tube in the flame and blow a bulb on the stem, finally drawing

off the end of the glass tube (Fig. 65) Marble is fixed in the test tube with a cork (Fig. 66), and the man immersed in hydrochloric acid, which is best put into a long piece of tubing, about  $1\frac{1}{2}$  in. in diameter. It is convenient to have a cork at the bottom carrying a tap, so that the acid can be renewed in the middle of the show, if necessary.

A face can be drawn with cellulose paint, which resists the action of acid for a short time

### 130. MUSICAL GELS

#### *I. Hepburn*

To prepare the gel, pour water-glass, diluted to 1.15 specific gravity, into an equal volume of 6 normal hydrochloric acid. With ordinary commercial water-glass, 1 volume added to 4 volumes of water will give about the right strength of solution. The solutions are quickly mixed and poured out into glass vessels of different sizes, half-filling them. Suitable vessels are thick test-tubes of various diameters, gas-jars, milk bottles, etc. After a few days, the vessels should vibrate musically if lightly held by the top and tapped smartly against the bench. The pitch of the vibration varies inversely as the diameter of the containing vessel.

### 131. METAL CRYSTALS IN GELS

#### *I. Hepburn*

The ordinary lead and tin trees are unsatisfactory for exhibition at a *conversazione* since a very small jolt destroys them at once. If the crystals are grown in silicic acid gels, the trouble does not arise and the crystals themselves are much better.

(a) *Tin Crystals* —The gel is obtained by adding water-glass solution of sp. grav. 1.15 to an equal volume of 6 normal hydrochloric acid, to which a few c.c. of stannous chloride have been added. The solution is poured into crystallizing dishes and allowed to set. When set, a piece

of zinc is pressed into the middle of the gel and tin crystals quickly grow. Twelve to twenty-four hours is quite long enough to allow. It is advisable to add varying quantities of stannous chloride to the gel, to ensure crystals of different sizes. It is possible to prepare the gels by making a pure silicic acid gel first, then adding stannous chloride solution, and allowing this to diffuse into it for an hour or two, afterwards pouring off, this may be the more effective method. The tin crystals will not last for more than a few days since the concentration of hydrochloric acid is great enough to dissolve them slowly.

(b) *Lead Crystals*.—More permanent crystals can be obtained with lead. The gel is obtained by adding water-glass solution of sp. grav. 1.06 (10 per cent.) to an equal volume of normal acetic acid, with a little lead acetate solution added to it. This is allowed to set in crystallizing dishes or small measuring jars, etc., and a piece of zinc is then added as before. Lead crystals take longer to grow than tin, and some of them should be started a good week before the exhibit is required. They are, however, permanent under these conditions.

(c) *Copper Crystals*.—A silicic acid gel is prepared with acetic acid as above, but with a little copper sulphate solution added. The gel is poured into a large test-tube. If the gel is covered with a 1 per cent. solution of hydroxylamine hydrochloride, small tetrahedrons of copper are formed in it after a week or two.

(d) *Lead Iodide Crystals*.—Very beautiful leaf-like crystals are obtained if the gel used in (b) is covered with a few c.c. of potassium iodide solution.

### 132. SILICA GEL DRIERS

The loose material can be bought. The Tell-Tale drier is coloured with cobalt chloride. This is not much by itself, but the addition of artificial flowers (roses are fairly easy), made with material impregnated with cobalt chloride, adds to the interest. If enough have been

prepared to give away specimens, so much the better. The made-up roses can be bought.

### 133. LIQUEFACTION OF AIR <sup>1</sup>

*R. W. Roberts*

The liquefaction of air may be readily demonstrated by means of the following experiment. A metal polish tin, A (Fig. 67), deprived of its paint and polished, is fixed to the piece of wood, B, as shown. The tin with its support is fitted fairly tightly into the transparent Dewar vessel, C, by means of an insulating ring of cotton-wool, D, fastened to A with a rubber band. Liquid air is poured into both A and C, the level of the liquid surface in C being about 1 in. below the bottom of A. On connecting A to an exhaust pump, it will be found that, as the pressure in A is reduced, a succession of drops will fall from the bottom of the tin. This clearly shows that the abstraction of heat from the air in C in the immediate neighbourhood of the tin has been sufficient to cause liquefaction. The effect may be enhanced by using, instead of a tin can, a vessel made of spun copper, with a round bottom.

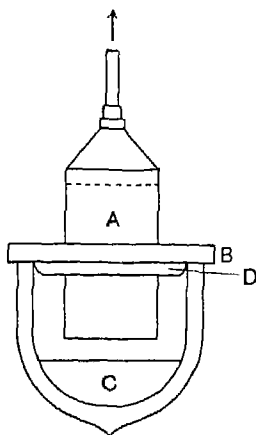


FIG. 67.

The experiment may be easily projected on to a screen.

### 134. "SOLID" METHYLATED SPIRIT

*W. H. Barrett*

A good conversazione demonstration. Make a saturated solution of calcium acetate (solubility 43.6 gm. in

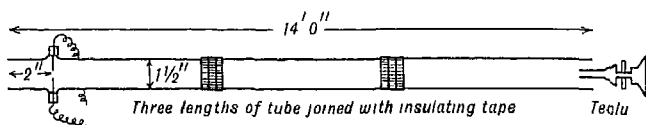
<sup>1</sup> From the George Holt Physics Laboratory, University of Liverpool

100 gm. water at  $0^{\circ}\text{C.}$ , 34.3 gm. at  $100^{\circ}\text{C.}$ ), and filter until quite clear. Pour 10 c.c. of this solution into one beaker and 90 c.c. of methylated spirit into another. Mix rapidly by pouring from one beaker to the other and back again. A white inflammable solid, rather like paraffin wax in appearance, is obtained. The volumes given above are for dehydrated industrial spirit.

### 135. EXPLOSION WAVE<sup>1</sup>

*W. H. Barrett*

The tube shown has a total length of 14 ft. The diameter of the tubing is 1 5 in. Three lengths may be joined with insulating tape. The tube is mounted in a



Stand

FIG. 68

wooden stand, as shown (Fig 68). The spark-gap is placed 2 in. from the open end

Bring the Teclu burner into the end of the tube and find by trial the length of time required to fill the tube with the explosive mixture. Remove the burner and pass the spark. To get a more rapid rate of explosion, put a cork loosely in the sparking-plug end after getting a mixture in which the flame travels slowly

<sup>1</sup> Modelled on the apparatus of Professor Bone.

## 136. DIFFUSION OF GASES

*G. C. Bachelor*

It is not suggested that there is anything new in the arrangement here described, but it forms a striking demonstration experiment and might usefully be staged at a conversazione.

The diagram (Fig. 69) should make the arrangement clear. It adds to the effect if the lamps are of different colours, say red and green.

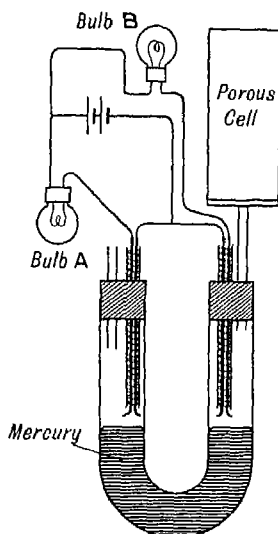


FIG. 69.

## 137. CHEMILUMINESCENCE

*I. Hepburn*

A pinch of o-amino-phthalic-cyclic hydrazide (obtainable from British Drug Houses) is dissolved in a litre of water. To this are added a few c.c. of potassium hydroxide and of hydrogen peroxide. When 100 c.c. of filtered bleaching powder extract are added, a brilliant blue luminescence is obtained

## 138. ELECTROTYPES

The diagram to be reproduced is drawn with a sharp-pointed instrument on sheet brass, thinly coated with wax. In actual practice, the diagram is then built up with liquid wax, to give printing depth. This, however, can be omitted in a demonstration. The wax is then polished with electrolytic graphite, using a soft brush. The waxed

plate is made the cathode, with pure sheet copper as the anode.

The following solution is recommended for the depositing tank :

Copper sulphate, pure cryst.	212 5 gm.
Potash alum, " "	12 5 gm.
Pure sulphuric acid	31 25 c.c.
Water . . . . .	1,000 c.c.

The solution for working must be cold, but not below 18° C. At the start, it is better not to have the cathode and anode too close ; begin with the plates 9–10 in. apart and decrease to 3–4 in. A current density of 5 ampères per square foot is used at the beginning, but after the surface is well covered, this may be increased to 20–25 ampères. The E.M.F. should never exceed 2 volts.

Special care should be taken to have a pure copper anode and to begin deposition very slowly.

### 139. "PHARAOH'S SERPENTS"

*R. E. D. Clark*

The author recently discovered accidentally that excellent "serpents" can be made by the following method

*p*-Nitroacetanilide (5 to 20 g) is added to five-ninths of its weight of concentrated sulphuric acid (1 mol.) in a small porcelain dish and the mixture is heated strongly on gauze, when it melts and a vigorous reaction begins to set in at about 200°. This is rapidly followed by a mild explosion, in which a large snake-like mass of carbon shoots out. In this way "snakes" a yard in length and as much as 5 in. in diameter can easily be obtained from a 100-c.c. dish. The snakes are black and sponge-like, and they are not nearly so brittle as those obtained from mercury thiocyanate.

Where a fume cupboard is available, it has been found that the experiment is admirably suited for lecture demonstrations. Acetic acid vapour is the main gaseous product evolved.

# 140. S-HOOK WITH MERCURY

*H. C. Palmer*

Glass tubes,  $\frac{1}{4}$ -in. diameter, are sealed at one end, filled with mercury and frozen in liquid oxygen. They are then transferred to an anvil and the glass cracked off. They are then "forged" to shape, being returned at times to the bath of liquid oxygen; care must be taken to cool the points of the tongs. The hooks will support a weight of several pounds.

# 141. MERCURY SPRAY

*I. M. Bankes-Williams*

A piece of plumber's cane, about 6 in. long, connects a mercury reservoir to the inside of a suitable bell-jar on the receiver of an air-pump. On working the pump, a fine spray of mercury is forced through the cane and is caught in a dish.

# 142. DENTAL AMALGAM

*H. C. Palmer*

Silver 75, tin 25, zinc 1. An alloy in these proportions is made and reduced to filings. To make the amalgam, 7 gm. of mercury and 5 gm. of filings are ground together in a mortar. This can then be pressed into a "cavity" made by boring a hole in a cork.

# 143. THE "MERCURY HEART" EXPERIMENT

*R. H. Adams*

Pour a drop of mercury, about  $\frac{3}{4}$ -in diameter, into a clock-glass, cover with water, add a few drops of sulphuric acid and stir in a crystal of permanganate, or a few grains of powdered potassium dichromate.



Fix a sewing-needle, mounted in a wooden holder, in a retort stand, and bring this up until the point touches the mercury in the positions shown in Figs. 70 and 71. When once started, the drops will continue to "beat" for a good time. The starting requires some patience; a change of needle may help, for no apparent reason. The needle must be steadied.

*A Suggested Explanation.*—The effect appears to be due

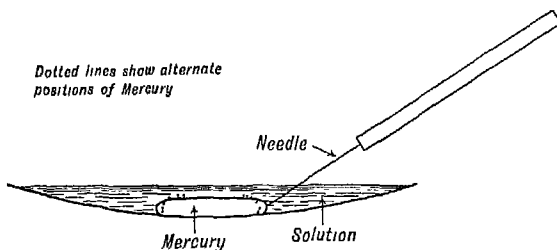


FIG. 70.

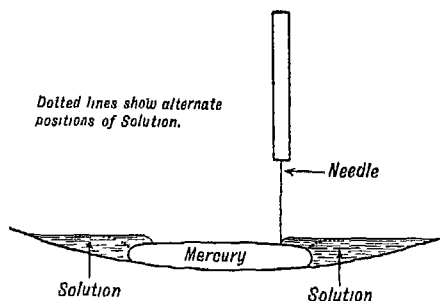


FIG. 71.

to the formation and polarization of a simple mercury-iron cell. When the needle touches the mercury, a current passes and a minute layer of gas ( $H$ ) is evolved at the mercury. Now the drop assumes such a shape that the sum of the gravitational and surface energies is a minimum. But the surface tension of a mercury-gas interface (466) is greater than for mercury-acid (320). Thus, when gas is formed, the surface energy becomes greater; hence the drop becomes more spherical, thus retreating from the

needle. Upon disappearance of the gas (greatly hastened by the depolarizing action of potassium permanganate), the surface tension falls again, the drop widens once more, touches the needle and the whole action repeated. This is confirmed by touching the drop with one end of a platinum wire, when no effect is produced; but if the other end of the wire is twisted round a needle, the phenomenon is observed whenever the needle is dipped into the acid.

Without potassium permanganate I could get no oscillation, but only a single quiver of the drop as the needle touched it. The magnitude of this quiver fell off with repeated contacts, and after four or five, it could not be detected, the cell evidently polarized rapidly. This effect was so small that I altogether failed to detect it until I had seen the phenomenon with potassium permanganate present. (B.M.N.)

#### 144. AUTOMATIC TITRATION

*H. C. Palmer*

A striking experiment illustrating the use of a photo-electric cell.

A photo-electric cell may be used to observe the end-point of a chemical reaction which causes the colour of an indicator to change. A suitable reaction is that due to Harcourt and Esson, where potassium iodide in sulphuric acid solution is oxidized by hydrogen peroxide, starch being the indicator. Thiosulphate can be added from a burette, and if a little excess of the thiosulphate be added, the slow oxidation and liberation of iodine do not immediately bring back the characteristic starch iodine coloration. Then suddenly, when the excess thiosulphate is used up, the whole solution darkens.

In order to make use of this reaction, a photo-electric cell is set up exactly as described in the *Science Masters' Book*, Series I, part 2, Appendix, page 241. The cell is

arranged to be activated by light from a galvanometer lamp, which is thrown in a parallel beam through the reaction vessel, preferably a small square glass tank. The reaction mixture should be mechanically stirred.

As soon as the mixture darkens, the cell returns to its condition of comparative insulation. This causes the triode valve to be negatively biased, and so its anode current falls and the relay in the anode circuit opens. An excellent relay for the purpose is the "Fultograph" relay, which may still be obtainable from disposal firms ;

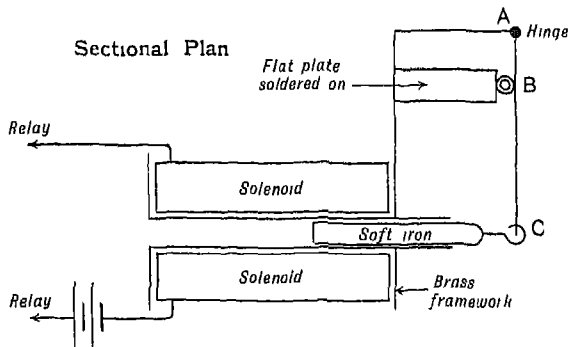


FIG. 72

it operates on a change of current of about one milli-ampère. Since the "Fultograph" relay is delicate, it is not politic to employ it directly to actuate the titration mechanism. Instead, it is used as a "master" relay and actuates a much more robust one as a "slave." This latter relay may be of any convenient form, a good one being a "bell relay." A mercury switch would also be excellent for the purpose, or any home-made device.

So far then, darkness in the photo-electric cell opens the sensitive relay and this causes the slave relay to open. It now remains to cause the slave relay to turn on a slow stream of thiosulphate solution when it opens. This is achieved by a solenoid device. When the slave relay closes, the solenoid "sucks in" the soft iron bar and actuates the lever ABC (Fig. 72), pulling with a mechanical

advantage of about *three* against the thin rubber tubing at B. This tubing may conveniently be bicycle valve rubber ; it is connected to a burette of *strong* thiosulphate solution and leads to the reaction vessel.

So long as the photo-electric cell is "dark," the solenoid is not taking current. Thiosulphate therefore is allowed to enter the reaction vessel. As soon as the starch iodide colour is discharged, the "master" and "slave" relays close ; the solenoid is magnetized and the rubber tube (Fig. 73) is pinched at B, thus cutting off the supply of

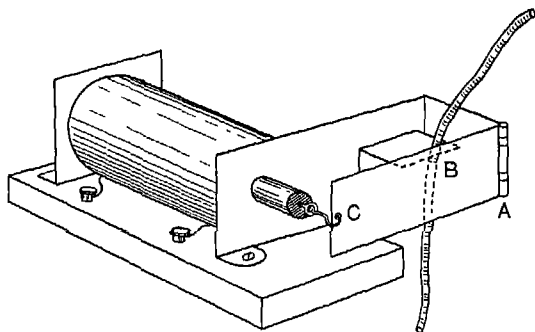


FIG. 73

thiosulphate. By arranging that the thiosulphate solution is strong, a slight excess can be added. It follows then that there will be a small interval during which this slight excess of thiosulphate is being used up. Once more the indicator changes and the whole cycle of operations is repeated.

The concentrations of the reagents should be chosen by trial-and-error methods, so that the relays operate at short intervals. It is possible to get the apparatus running so that the "end-point" is reached and the relays operate scores of times without any human intervention.

Other reactions may be employed, using transparent coloured indicators. It may be then necessary to insert a light filter in the light beam in order to make the response of the photo-electric cell effective.

Practical details of Harcourt and Esson's experiment are given in Fenton, 1910 edition, pp. 141-2.

The automatic titration apparatus was first devised, so far as I know, by Mr. P. T. W. Baker, when a boy at Oundle School. It has subsequently been modified in details.

#### 145. DYES

Methyl orange is an easy preparation, malachite green is more difficult, but several intermediates can be shown; also a steam distillation can be introduced in order to have something "working."

#### 146. DYEING

Home dyeing is fairly common; nevertheless, the processes seem to interest visitors. A background of dyed materials—pieces of old silk and skeins of wool—makes a bright show. Some supervision is needed to prevent clashing of colours

#### 147. DYEING WITH NATURAL PRODUCTS

Many natural dye-stuffs, such as Persian berries, fustic chips, cochineal, cudbear, indigo and even woad (sometimes) can be bought from dealers. They are used for dyeing and tinting hat shapes. This exhibit is all the more interesting because the original materials look so unpromising. Useful information on this subject will be found in *Use of Vegetable Dyes for Beginners*, by Violetta Thurstan, The Dryad Press, Leicester; price 2s 6d.

#### 148. ADSORPTION AT INTERFACES AND COLOUR CHANGES OF DYES

*A. J. Mee*

It was discovered by Deutsch (*Ber.*, 1927, **60**, 1036; *Z. physikal. Chem.*, 1928, **136**, 353) that a displacement

of the equilibria between molecular and ionic species in certain dye solutions took place when they were shaken with an indifferent liquid, such as benzene, toluene, hexane, or carbon tetrachloride, the effect being entirely due to the preferential adsorption of one or other of the species. The displacement of the equilibrium results in a change in colour, so that, for instance, when an acidified solution of malachite green is shaken with benzene, the colour changes from a greenish-brown to green. The more vigorous the shaking, the deeper is the green colour, since the droplets are smaller, a larger interface is present, and the adsorption is greater. When the vessel containing the mixture is allowed to stand, the original colour returns with the separation of the two layers, and the process may be repeated indefinitely. Some of Deutsch's experiments are quoted by Freundlich in the Second Liversidge Lecture, delivered before the Chemical Society in 1929 (*J.C.S.*, 1930, 164), and reference should be made to this paper for the theory of the colour change. The experiments described below, which make very effective demonstrations for a conversazione, have been obtained largely from this source, though the methods of performing them are described here in greater detail than in the paper referred to, and in some cases, new experiments are mentioned. The dyes employed are in common use as indicators and stains, and may all be purchased from the British Drug Houses Ltd. at a reasonable cost.

1. *Malachite Green*.—Take about 50 c.c. of a dilute solution (0.01 per cent.) of malachite green in water in a small reagent bottle (125 c.c.). Add a few drops of strong hydrochloric acid, until the colour changes to a greenish-brown. Now add about 50 c.c. of benzene, carbon tetrachloride, toluene, or xylene. On shaking the bottle vigorously, the emulsion formed becomes green in colour. When the bottle is allowed to stand, separation into two layers takes place, and the aqueous layer once again has its original colour. The colour fades on keeping, but it is quite satisfactory for a few days.

It must be mentioned that the colour of the aqueous solution varies somewhat with the dilution and the amount of acid added. No doubt other indifferent liquids may be used in place of those mentioned above.

2. *Xylene Cyanol FF* (oxidation-reduction indicator) — This dye is soluble in water to give a blue solution. Prepare a very dilute (0.01 per cent) solution and add one drop of strong hydrochloric acid, which turns the colour green. On shaking with benzene, the colour changes to a beautiful turquoise blue. When the bottle is allowed to stand, separation occurs, and the aqueous layer becomes green again. The dye is insoluble in benzene, so that after separation, the upper layer is perfectly colourless. This experiment is probably the most striking in this class, since the colours are so beautiful and the inert liquid is not coloured by the dye.

#### *Acidimetric Indicators.*

In order to obtain colour changes with these indicators analogous to those obtained with the dyes just mentioned, the *pH* of the solution must be very carefully adjusted. For this purpose, the use of a buffer solution is strongly recommended. Preferably one of the Universal Buffer Mixtures should be used, such as that of Britton and Robinson (*J C S.*, 1931, 1456), or that of Prideaux and Ward. Both are obtainable from the British Drug Houses Ltd., ready mixed. Solutions of any definite *pH* are readily obtained from these mixtures by the addition of a definite volume of standard sodium hydroxide or hydrochloric acid.

It is difficult to obtain the colour changes if alcoholic solutions of the indicators are employed, probably because the surface tension is different from that of an aqueous solution, and there is a certain miscibility of the benzene and aqueous-alcoholic layers which prevents good results.

*Bromothymol Blue.*—When a small quantity of this indicator is dissolved in a mixture of distilled water and tap water (*pH* about 7.4), and the mixture is shaken with

carbon tetrachloride or benzene, the colour of the emulsion becomes yellowish-green. The more vigorous the shaking, the more yellow does the colour become. On standing, the blue colour of the aqueous layer returns, and the indifferent liquid is left quite uncoloured. The B.D.H. indicator made up for capillator use answers very well for this experiment.

### *Fluorescent Dyes.*

These show some of the most vivid colour changes of the whole series and are most interesting from many points of view.

*Rhodamine 6G* (adsorption indicator).—When a dilute aqueous solution of the fluorescent dye, rhodamine 6G, is shaken with benzene, or carbon tetrachloride, the fluorescent red colour changes to a bright pink. On settling out, the original colour is restored to the aqueous layer.

Rhodamine 6G is the salt of a base which is difficultly soluble in water, but is much more soluble in benzene, in which it dissolves with a yellow colour. If the solution of the base in benzene is shaken with water, the phenomenon of the colour change is observed from the reverse side, so to speak, the coloured droplets of benzene being dispersed in the water. When this is done, the whole liquid becomes bright pink.

To carry out this experiment, prepare a dilute aqueous solution (0.01 per cent.) of rhodamine 6G, and to 10 c.c. of it, add 5 c.c. of bench sodium hydroxide solution (about 10 per cent.). Shake with 15 c.c. of benzene for a few seconds. The addition of the sodium hydroxide liberates the base of rhodamine 6G, which is then extracted by the benzene, which becomes coloured yellow. Separate the benzene solution. Shake some of it with an equal volume of water. The emulsion becomes red, and on separating out again, the benzene layer is still yellow, and the aqueous layer slightly pink, owing to the formation of the red betaine form of the base in the aqueous phase. A



pink precipitate is sometimes formed at the interface on standing.

Now pour some of the benzene solution of the base on to a small heap of dry Calais sand, powdered glass, or quartz. The heap becomes bright red, owing to the adsorption effect.

Another very effective experiment is to dip a piece of filter paper into the yellow benzene solution. The filter paper becomes red as soon as it is placed in the solution. Or, the benzene solution may be dropped on to a filter paper. As each drop falls, a red stain is produced. The effect is not due to the evaporation of the benzene, for the colour is red whilst the paper is still wet.

#### 149. CHEMICAL FLAVOURS AND PERFUMES

A very economical method of putting these up was indicated in First Series, Part 2, p. 247. The following list will be found useful:

*Alcohols*—Benzyl alcohol, geraniol, citranellol, terpineol, santalol

*Aldehydes*—Citral, benzaldehyde, phenylacetic aldehyde (Hyacinth), cinnamic aldehyde, anisic aldehyde (Aubepine), vanillin, heliotropin.

*Ketones*.—Camphor, ionone (Orris root butter).

*Phenols*.—Thymol, anethol, safrol, myristicin (Nutmeg).

*Esters*.—Methyl benzoate (Niobe), methyl salicylate (Winter Green), amyl acetate (Pear), amyl valerianate (Apple), ethyl butyrate (Pineapple), amyl salicylate (Trefoil), linalyl acetate (Bergamot), geranyl acetate, benzyl acetate (Jasmin), bornyl acetate (Pine oil), methyl anthranilic acid, methyl ester (Mandarin Orange).

*Lactones*.—Coumarin (Tonquin Bean).

*Nitro-Compounds*.—Nitrobenzene, trinitro-tert-butyl-toluene (Musc Baur).

## 150. BLENDED SCENTS

	I Eau de Cologne	II Eau de Cologne	Lavender Water	Epsom Downs Perfume	Paris Perfume.
	c.c	c.c	c c	c c	c c.
Absolute alcohol .	100	100	100	—	100
Oil of bergamot .	0 88	1 00	0 88	0 35	0 90
Oil of lavender .	—	—	1 78	—	—
Oil of lemon . .	0 52	0 50	—	—	0 40
Oil of mace . . .	—	—	—	0 36	0 20
Oil of neroli . .	0 30	0 20	—	—	0 30
Oil of rosemary .	0 04	0 05	0 04	—	—
Oil of organum .	0 04	—	0 04	—	—
Extract of jasmine	—	—	—	24 0	0 52
Extract of rose	—	—	—	32 0	—
Extract of tuberose .	—	—	—	16 0	—

These put up in scent sprays with samples of the original oils and extracts make a very popular item

## 151. SIMPLE PHENOL-FORMALDEHYDE RESIN

Phenol (cryst) . . . . .	100 parts by weight
Formaldehyde (40 per cent aqueous solution) . . . . .	80 " " "
Ammonia (0.880) . . . . .	8 " " "

Heat the above mixture in a flask with reflux condenser and continue boiling for twenty minutes. At the end of about eight minutes, there should be separation into a watery and an oily layer. After boiling for the prescribed time, it is advantageous to transfer the product from the flask to an open vessel—a metal saucepan is quite suitable—and continue heating until a test sample, on cooling in water, is found to be hard and brittle. The resin is then poured out in a flat tray to cool.

This process yields a spirit-soluble stoving resin.

## 152. PLASTICS

*H C. Palmer*

Another quickly made resinoid can be got by heating together 40 per cent. formalin and resorcinol in the presence of a little caustic alkali. The results are startling.

For demonstrating the actual moulding, it is advisable to get the powder from the manufacturers. The pressure should exceed 1 ton per square inch. Small mouldings can be made by using a modified vice or screw jack.

Owing to difficulties of temperature control and inequalities of pressure, the results are often disappointing. A very useful "thermoplastic" which is far easier than bakelite for amateurs to mould is benzyl cellulose (Imperial Chemical Industries).

### 153. GUN COTTON

*G. H. Locket*

Run five parts of concentrated  $\text{H}_2\text{SO}_4$  (by vol.) into four parts of concentrated  $\text{HNO}_3$ . Cool and immerse a large tuft of cotton-wool. Leave for an hour. Drop into water and wash until the wool no longer tastes acid. It is a good thing to tease out the wool at the washing stage, but it should not be squeezed. Dry at room temperature (two to three days)

To show the relative rates of burning of nitrated and ordinary cellulose have test-tubes, one containing a tuft of cotton-wool and the other gun cotton. The demonstrator lights them both on a piece of asbestos, side by side.

### 154. PAPER FROM WOOD

*G. H. Locket*

File up wood with a big rasp (sawdust has too short a fibre). Boil for an hour with 5 per cent.  $\text{NaOH}$ . Wash on a 40-mesh copper gauze, fixed to a wooden frame and arranged over a sink. Squeeze out the dark pulp and put it into a large beaker. Admit chlorine (bleaching powder and  $\text{HCl}$  best for this) until no further change. Add 1 per cent.  $\text{NaOH}$ , leave for a quarter of an hour, wash, squeeze and chlorinate as before. Go on repeating

the process until the pulp is quite white <sup>1</sup> (The thing can be shown in various stages in the show )

Suspend the pulp in water in a wooden box, lined with pitch, bring gauzes under it and fish out thin layers of it. Dry these in an oven ; they can then be detached, making a very tolerable blotting paper. (Sizing has not, so far, been successful.)

## 155. PIGMENTS

*H. C. Palmer*

*Manganese Brown.*—To manganese chloride add sodium hydroxide, also ammonia and hydrogen peroxide. Filter, wash, dry.

*Zinc White.*—Zinc chloride and limewater.

*Scheele's Green.*—Mix solutions of copper sulphate and sodium arsenite and add potassium carbonate solution until the full colour is obtained.

*Viridian Green.*—Fuse 8 parts boric acid with 3 parts potassium bichromate. Grind, wash, dry.

*Freeman's White Lead.*—Precipitated lead sulphate.

*Venetian Red.*—Ferric oxide from ferrous sulphate by ignition.

*Cadmium Yellow.*—Cadmium sulphide.

*Lemon Yellow.*—Barium chromate.

*Cobalt Blue.*—Ignite aluminium oxide with cobalt nitrate. Wash and dry.

*Crimson Lake.*—Extract cochineal with boiling water. Add a solution of potash alum, followed by potassium carbonate. Filter off the lake.

*Chrome Orange.*—Make potassium chromate solution alkaline with lime water and add lead acetate solution until the full colour is brought out. Filter.

*King's Yellow.*—Arsenic sulphide.

*Chrome Green.*—Ignite ammonium bichromate and grind.

<sup>1</sup> The bleaching process was devised by Mr. A. G. Pollard, Imperial College of Science.

*Chrome Yellow*.—Lead chromate.

*Prussian Blue*—A ferric salt and potassium ferrocyanide.

*Turnbull's Blue*—A ferrous salt and potassium ferricyanide.

Many other lakes and powdered vat dyes will suggest themselves. The products can be mixed with gum-water and painted on cards.

## 156 LUMINOUS PAINTS

*H. C. Palmer*

There are several recipes for luminous paints given in Partington, 1921 edition, p. 877. We have tried these at Oundle and have had success with them.

For demonstration purposes, we paint them on cards and irradiate them with a quartz mercury vapour lamp, or other suitable source. When freshly irradiated, they glow intensely, and the diminution of luminosity with time is quite marked.

## 157. ARTIFICIAL SILK—VISCOSE

*H. C. Palmer*

Though this may be prepared from wood pulp, it is better, under laboratory conditions, to use cotton-wool.

Cotton-wool is steeped in 18 per cent caustic soda solution at room temperature for an hour or two, and then compressed to expel excess alkali. The cotton-wool, so soaked, weighs about three times as much as when dry.

The "alkali cellulose" is now ready for treatment with carbon disulphide, but the results are better if the alkali cellulose is kept in contact with air for two or three days (broken up into small pieces).

Cellulose xanthate is now made by slowly "churning" the product with carbon disulphide. This is conveniently

done by enclosing the reaction mixture (with a slight excess of carbon disulphide under laboratory conditions, to allow for volatility) in the apparatus illustrated

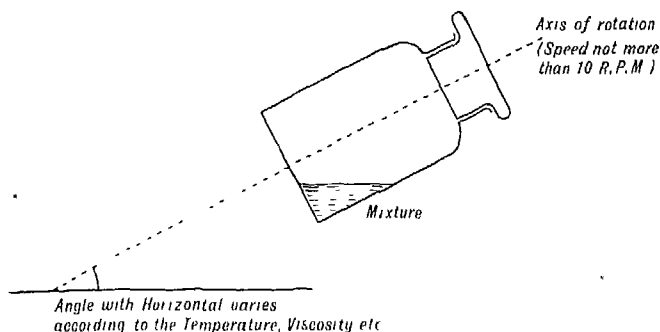


FIG. 74.

(Fig. 74) for three hours, at from 24°–27° C (It works well enough at room temperature.)

When the reaction is evidently complete and all threads of unchanged cellulose have vanished, the mixture is thinned out with dilute caustic soda solution (0.25 per cent.) until it contains 7–8 per cent. of cellulose and 6–7 per cent. of caustic soda, and churned again

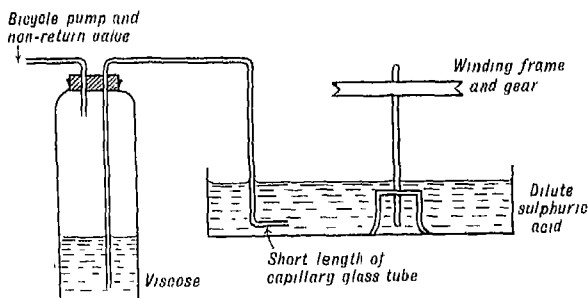


FIG. 75.

for a while. It should then be left to age for four to five hours at room temperature.

*Spinning.*—For demonstration, single threads are spun from glass capillaries, of about  $\frac{1}{16}$  mm. diameter, into a

bath of dilute sulphuric acid and sodium sulphate (8.5 per cent  $\text{H}_2\text{SO}_4$ , 15 per cent.  $\text{Na}_2\text{SO}_4$ ), and wound on glass frameworks dipping under the acid. When sufficient thread has been made, the framework of glass is transferred to a water-bath, and thence to a solution of sodium sulphide, to remove precipitated sulphur (We usually omit the sulphur-bath.) Finally, the thread is washed and dried.

### 158 SPINNING ARTIFICIAL SILK

*G. H. Locket*

The device shown in Fig. 76 is very useful. A is a home-made pump, with a valve made of a bored cork

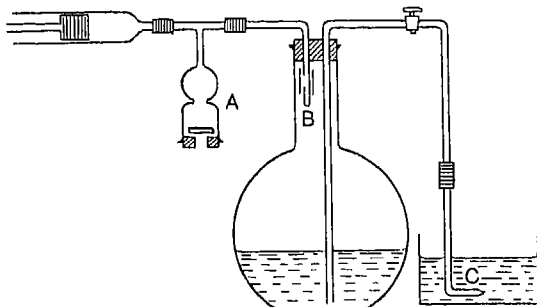


FIG. 76.

and a loose disc of rubber to lie over the hole. B is a closed glass tube, with a hole in the side covered by rubber tubing—an imitation of a bicycle tyre valve. C is a removable jet, joined on by pressure tubing. It is useful to have several jets of different diameters.

### 159. CELLULOSE ACETATE

*H. C. Palmer*

The making of this cellulose ester is described on page 289 of Sherwood Taylor's *Organic Chemistry*. It may be spun into threads in the manner there described.

A striking experiment is to immerse glass test-tubes in an acetate solution of cellulose acetate and then dry them off in a current of *dry* hot air. If the air is not dry, the film is not transparent. Shapes so formed may be separated from their formers by soaking in water for about an hour. A small amount of triphenyl phosphate may be added to solutions of cellulose acetate as a "plasticizer."

## 160. FROTH FLOTATION

*H. C. Palmer*

Use a miniature butter churn (obtainable from iron-mongers), or a geared paddle in a rectangular glass jar.

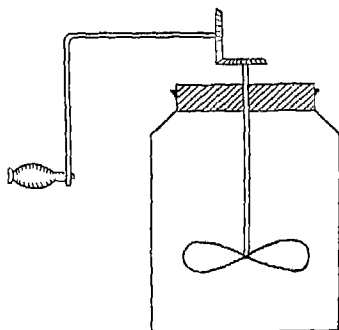


FIG. 77

The ore is powdered in a mill. Add some "froth former," such as creosote, xanthates, etc.

## 161. HYDROGENATION OF OILS

*H. C. Palmer*

The catalyst. Prepare pure nickel formate and reduce this in a current of pure hydrogen<sup>1</sup> at the lowest tem-

<sup>1</sup> At Oundle we sometimes use electrolytic hydrogen from a battery of cells—iron cathodes in caustic soda solution.



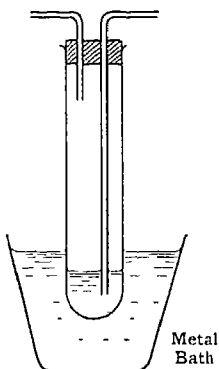


FIG. 78.

perature at which reduction will take place. Heat the formate in a metal bath as shown

Pour the reduced nickel directly into olive oil and hydrogenate at about  $200^{\circ}$  C. or under. When the mass solidifies, filter hot into specimen tubes.

## 162. FOAM FIRE EXTINGUISHER

*G. H. Locket*

Take two aspirators, one containing saturated sodium bicarbonate (with 1 or 2 gm. of saponin or liquorice extract), the other a solution of

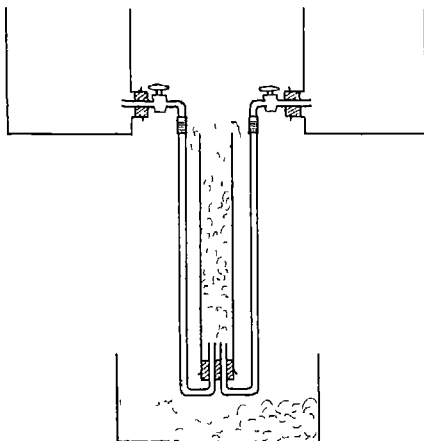


FIG. 79.

aluminium sulphate, with a little sulphuric acid. Connect these to the base of a piece of wide tubing, which fills with foam and overflows whenever the taps are turned on.

## 163. OSMOTIC PRESSURE

*F. Farbrother*

A piece of old drum parchment is thoroughly softened by soaking in water and tied tightly with pack thread over the rim of an enlarged thistle funnel head. A 50 per cent. sugar solution is made up with water coloured with magenta. This is poured into the thistle funnel head. Provision is made for an osmotic pressure column of 15-20 ft.

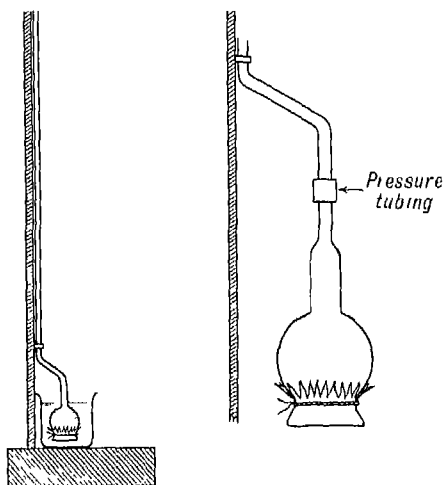


FIG 80.

 164. HOOKE'S EXPERIMENT. "FLUID" PROPERTIES OF SAND<sup>1</sup>

*Donald H. Bellamy*

To illustrate this, obtain a wooden box, about 12 in.  $\times$  6 in. and about 8 in. deep. Fill this almost to the top with very fine *dry* sand. Mount the box so that

<sup>1</sup> See *Concerning the Nature of Things*, Sir William Bragg, p 26, 1927 Ed

it can be shaken backwards and forwards and up and down. This can be done by mounting the box on two eccentrically mounted wheels. When shaken, the sand appears to be "running". Place a light object, say, a toy celluloid duck, under the sand. Almost immediately it will rise to the top of the sand and ride on the surface. Heavy objects, such as a piece of lead or a coin, will be found to sink to the bottom of the box.

### 165. FORMATION OF WATER DROPS

*J. M. Bankes-Williams*

Water is dropped from a dropping funnel into olive oil. At room temperature, the drops are perfectly spherical and fall slowly, without the deformation so often seen when water and aniline at different temperatures are used.

### 166. BALLISTIC BALANCE

*J. M. Bankes-Williams*

For measuring the velocity of a 0.22 bullet. The type of balance mentioned in Watson's Physics is used. A weight of 8 lb is suitable. The central pillar should have  $\frac{1}{8}$ -in steel plates at back and sides, and the wooden block should be made detachable so that it can be replaced when necessary. For demonstration purposes, a pivoted pointer rests against the end of the balance, with a piece of mirror fixed to the pivot. A spot of light is reflected on to a translucent screen, which can be marked off direct in velocities. The balance also provides a profitable specialist experiment.

### 167. MOTOR-CARS—STEERING AND SKIDDING

*J. P. Stephenson*

Scale model motor-cars can now be obtained from the shops. The front wheels can be modified to give true

Ackerman steering; most people will be interested in this. A Meccano differential can be fitted to the rear wheels. Simple arrangements can be made to lock any wheels. The effect of locked wheels on skidding can then be shown by propelling the car down a smooth table with a vigorous push of the hand. With the back wheels locked, skidding always occurs; with the front wheels locked, this rarely happens.

## 168 HYDRAULIC RAM

*K. C. Barnes*

The diagram (Fig 81) shows a very successful model set up in the Bedales laboratory. The wider glass tubes are  $1\frac{1}{8}$  in. diameter and the narrower ones  $\frac{3}{4}$ -in. All the

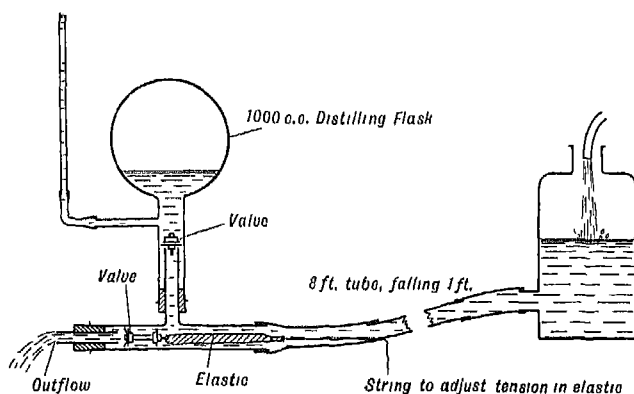


FIG. 81.

joints are made with wide cut-sheet rubber tube. The valves are made with Meccano-flanged wheels, faced with rubber discs cut from a car tyre inner tube. The upper operates by its own weight while the lower one is controlled by an elastic band and string. The ends of the tubes against which they fit are ground flat on a stone. The only difficulty met with in making the model was in sealing the T-joint into the wide tube; the joint

must be annealed carefully so that the shocks experienced during working do not start a crack.

### 169. INFRA-RED RADIATION

*J. P. Stephenson*

An electric bowl fire element is a suitable source of infra-red, and a small radiometer is a satisfactory detector. A sheet of thin ebonite is a satisfactory filter since it cuts off the visible rays. The focusing effect of metal mirrors can be observed; the ebonite should be formed into a cylinder surrounding the heater. The diathermancy of different substances can be shown. In this experiment, the heater is placed in a box with an ebonite window, and various substances are interposed between it and the radiometer.

A small radiometer will spin round quite rapidly when outside the red of the spectrum of an ordinary glass prism.

### 170. JUMPING DISCS

*J. P. Stephenson*

Welded brass-invar strip, also discs stamped out of the strip so that they are slightly concave on the brass side, can be bought from apparatus dealers. By pressing between the finger and thumb, the discs are flattened, and when the brass side is placed on a cool surface, the discs suddenly revert to their original shape and jump 2 or 3 ft. into the air.

### 171. MIRROR DRAWING

A (Fig. 82) is a piece of paper, with a large star drawn on it, B is a plane mirror and C is a piece of cardboard, or three-ply wood, which serves to hide the hand and the object to be drawn, but allows a person standing up to see the image of the star in the mirror. The visitor is

given a pencil and is asked to trace over the star, looking at the image in the mirror.

Turning corners is, of course, difficult, for the mirror interferes with the normal co-ordination between hand and eye. In strict circles, of course, the task is to be done against time. The necessary stars can be quickly made by drawing one and pricking through the points with a needle on to several sheets of paper placed beneath.

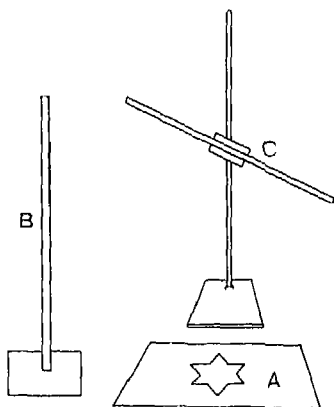


FIG 82.

## 172. DIFFRACTION PATTERN WITH HAT PINS

*I. M. Bankes-Williams*

A very effective pattern can be formed by using two pins, of diameter 3 mm. They are mounted so that the heads just touch, about 3 ft. from a 0.5-mm. hole, drilled in a piece of brass. The image can be observed with a 10-cm. lens. It saves time to put a piece of card, with a hole cut in it (about 2 to 3 mm. in diameter), in the position the observer's eye should take.

## 173. VANISHING PICTURES

*Eric Rogers*

Most red inks and paints on white paper are invisible when illuminated through a red filter. For green, the following works well. For ink. ordinary Stephens's green ink, diluted a little with water. For filter. Wratten blue-green, No 75, supplied by Kodak, Ltd.

## 174. ELECTION POSTER

*Eric Rogers*

The following works well with red and green filters. Prepare a poster marked, say, VOTE FOR JONES, in black indian ink. Exhibit this poster in blue-green (or green) light, and explain that a rival proceeded to alter it. With a large brush, dipped in red ink, cross out "FOR JONES" and add, say, "DON'T" at the beginning. Explain that Jones, however, viewed matters in a rosy light, and change to red illumination.

## 175. OPTICAL ILLUSIONS

*J. P. Stephenson*

(1) A small cubical cardboard box can be made which will just hold an ordinary laboratory glass cube. Holes are made with a cork borer in two adjacent faces of the empty box. That light can pass through these holes can be verified by holding the box up to a window or lamp. When the transparent glass cube is put inside, one can no longer see through these two holes

(2) A similar experiment on total reflection can be done with a glass cube having one face painted with white enamel. If this is placed, white face upwards, over a sixpence, the coin will "disappear," at least, it cannot be seen through the sides of the cube.

(3) A glass tube, about 6 in. long, has a piece of glass rod sealed to one end and extending about 3 in. into the tube along the axis. The end of the rod should be rounded. If the tube is now half-filled with a liquid of the same refractive index as the rod and sealed up, the rod disappears when covered by the liquid. A suitable mixture can be made by adding carbon tetrachloride gradually to  $\alpha$  bromnaphthalene

(4) An interesting illusion, marketed by Max Kohl, consists of a  $60^\circ$  prism with one vertical edge ground away

and patterns blasted on the adjacent faces. On looking into the front of the prism, one can see a beautiful crystal vase of flowers. The effect is again one of total reflection and can be imitated by cracking away glass from one edge of a prism with a pair of pliers.

#### 176. VELOCITY OF LIGHT IN AIR AND IN WATER

*J. P. Stephenson*

The fact that light travels more slowly in water than in air can be shown by setting up some interference fringes system (Fresnel's is probably the easiest), on an optical bench. If an arc or mercury vapour lamp is used, these can be projected on a screen. If a tank, with plate glass ends, is now interposed between source and screen and the fringes are focused on one end of it, the difference in width of the fringes which have passed through the water shows that this part of the light has been slowed down

#### 177. GRASSOT'S STAR

*J. P. Stephenson*

If three Geissler tubes are fixed at  $120^\circ$  on the face of a disc and rotated by the whirling table, very pretty patterns will be observed. Interesting stroboscopic effects can be produced by altering the tension in the spring of the induction coil hammer. Grassot produced further variations by including a microphone in the circuit, so that the pattern seen depended on the speech entering it

#### 178. MIRAGE · BENDING OF A BEAM OF LIGHT

*J. P. Stephenson*

The phenomenon of a mirage can be imitated in a glass trough. The smallest trough that can be used is



about 9 in.  $\times$  3 in.  $\times$  4 in.; a larger trough is better. Equal quantities of water and alcohol should be slightly coloured with fluorescein. The trough should be half-filled with alcohol, when a similar amount of water is introduced by a funnel, the surface of separation can be clearly seen. After a few days, the alcohol will have diffused into the water and the refractive index will vary with the depth. The same effect can be produced immediately by very careful stirring. When a beam of light is shone at an angle of about 20–30°, it encounters a medium of less and less refractive index, until it is totally reflected and passes through the liquid in a beautiful curve.

## 179 LUMINOUS CASCADE

*J. P. Stephenson*

In this arrangement, a lamp is used as illuminant and the water runs from a closed cylinder, one end of which is formed by a lens or lenses. The apparatus can be connected directly to the tap, and colour effects can be produced by interposing gelatine filter between the lamp and lens, a slit being provided for the purpose.

A brass tube, 2 in. diameter inside and 4 in. long, is closed at one end by a brass disc, fitted with a short jet about  $\frac{1}{4}$ -in. internal diameter. A 5-cm. focus double-convex lens is fixed 1 in. inwards from the other end, being cemented there by shellac. This then forms a watertight compartment into which water from the tap can flow if a side tube is fitted as in the drawing. A similar lens is held in contact with the first by a circular piece of stout brass wire or a rim of brass.

The lamp is held opposite the lenses in an outer sleeve fitting over the tube, and the bulb, a 12-volt car bulb, or a 12-volt, 24-watt G.E.C. projector bulb, fits into a standard small lampholder soldered to the end of the tube.

The only difficulty in the construction is the fitting

of the lens to make a watertight joint. If a small ring of brass is cut from a piece of 2 in. tube, it can be made to fit inside the lens tube by cutting a little from its circumference with a hacksaw. If it is pushed inside the tube, it will serve to hold the lens whilst it is being sealed in position. This sealing can be done in several ways, but in this case, shellac was used. It was first softened and then rolled with the fingers until an annular ring of about 2 in. diameter was formed. This was laid

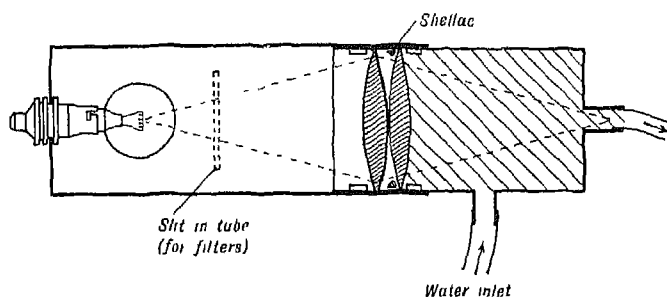


FIG. 83.

on the lens inside the tube, and melted there with a hot poker. When it had set, the other lens was fitted as mentioned.

If the stream of water is turbulent, it can be made to flow smoothly by fitting a piece of rubber pressure tubing over the nozzle.

## 180. PROJECTION KALEIDOSCOPE

*J. P. Stephenson*

This is rather an old-fashioned arrangement for producing on a screen those patterns usually seen by direct vision. It can be made quite cheaply in the following way.

A piece of cardboard tube, 10 in. long, 2 in. diameter, two pieces of plane mirror, 9 in.  $\times$  1 $\frac{3}{4}$  in., and a piece of card the same size, two convex lenses, 20 cm. focus

and 2 in diameter, are the principal materials required. Fit the mirrors inside the tube by using the strip of card; the fact that they are all the same size will ensure that the angle between the mirrors is  $60^\circ$ . From a circular piece of tinfoil cut a V as shown in the diagram, and slip this inside the tube at one end to act as a mask, and to prevent light shining down the tube behind the mirrors; pieces of passe-partout stuck on the lens will answer the same purpose. Now close the ends of

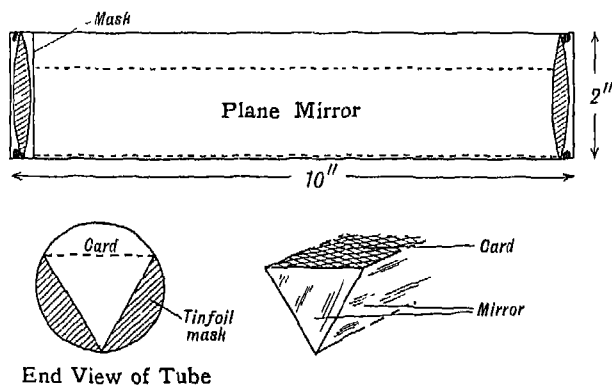


FIG. 84.

the tube with the lenses; they will stay in the tube if this is a good fit; otherwise soft wax must be used.

This arrangement takes the place of the objective of an ordinary lantern, and the various objects used to produce patterns are put near the condenser. Some difficulty will, however, be experienced in getting even illumination on the screen. It is best to fix some object (e.g. a sheet of clear glass with a few dabs of paint on it) near the condenser and then to move the kaleidoscope about in front until an image is formed on the screen. The end of the tube nearer the condenser should be tilted down a little so that the light, after passing through the first lens, falls on the mirrors before passing completely through the instrument.

As objects it is suggested that pieces of bent copper wire, string and coloured gelatine be used.

## 181. FOOT-CANDLE METER AND ILLUMINATOR

*J. P. Stephenson*

A Weston or a Sharpe's foot-candle meter shows the intensity of illumination falling on any surface. An interesting exhibit on illumination can be worked out. Lamps of different wattage can be used and their powers compared with a standard candle. The effect of different reflectors and various colours is also interesting.

## 182. RESONANCE TUBES

*I. M. Bankes-Williams*

A sufficient number of test-tubes, of 1 in. diameter, to make a complete octave and a few extra notes is needed. A motor-driven blower is essential and a flattened nozzle is more effective. In the hands of a musical demonstrator, a great variety of tunes can be obtained, and the audience may safely be allowed to handle the apparatus.

## 183. SMOKE DISPERSAL

*I. M. Bankes-Williams*

A box is made, about 8 in. high, 8 in. wide and 3 in. deep, with a glass back and front. Through an ebonite plug in the lid (which is insulated from the body) is passed a piece of  $\frac{1}{4}$ -in. brass rod, tapering to a fine point. The rod is connected to the positive of a Wimshurst. The box is filled with smoke and the Wimshurst worked. The smoke rapidly disperses and condenses on the sides and bottom.

## 184. SMOKE ABATEMENT

*H. C Palmer*

The Lodge-Cottrell method of electrostatic precipitation of fumes is quite readily demonstrated on the laboratory scale.

A tall vertical tin tube (Fig 85) has a diameter of about 2 in. A carefully insulated central wire hangs along the vertical axis of this tube. By means of an induction coil, a voltage is maintained between the two conductors just short of sparking. The apparatus should be about 3 ft. high. When in good running order, this apparatus will deposit ammonium chloride fumes. This can be verified by interrupting the action of the coil

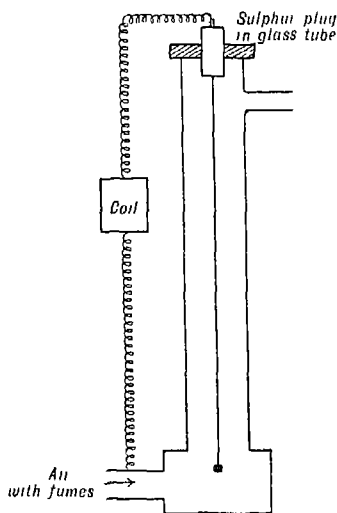


FIG. 85.

It should be easily possible to control the operation of this apparatus by

photo-electric cell and a system of relays arranged so that the operation occurs only when "smoke" is ascending the shaft.

## 185. MAGNETIC RECORDING

*W. Llowarch*

The output of an amplifier is taken to a small electro-magnet, between the poles of which a steel wire, or tape, is moving. A magnetic record of the sound is thus obtained. To play back the record, the electro-magnet is used as a pick-up and connected to the input ter-

minals of the amplifier. Results of some interest, illustrating the principles, but not of course of high quality, may be obtained by recording on piano wire with simple apparatus

About 20 ft. of piano wire, of 24 S.W.G., is made into



FIG. 86.

an endless loop. This is done by filing away the ends of the wire obliquely as in Fig. 86, and then joining them together with silver solder. The joint is filed down until it is uniform in thickness with the remainder of the wire. The loop is passed over two wooden pulleys, of 8 in. diameter, and one pulley is driven by a motor so that the wire travels at a speed of about 4 ft. per second.

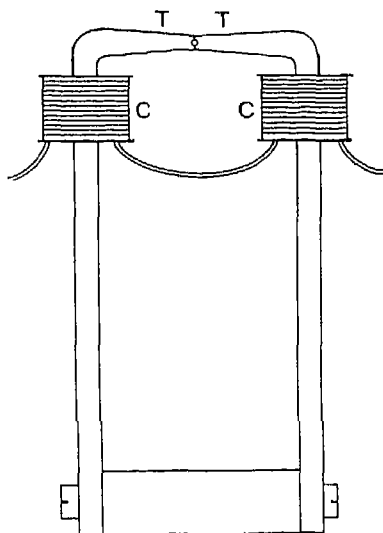


FIG. 87.

The electro-magnet is built up of soft iron, as shown in Fig. 87. It is approximately 2 in. high. It would be equally satisfactory if bent up out of a single strip. The pole tips are filed to knife-edges. A small "nick" in each knife-edge serves to locate the wire. Rubber bands, or a light spring, may be used to maintain contact between the pole tips and the wire if there is not sufficient spring in the iron strip. The coils, C C, may be wound on bobbins, or the poles may be wrapped with insulating tape and

the wire wound direct on to this. Each coil contains forty turns of No 30 enamelled copper wire

The pole tips, T T, are slightly "staggered," making contact with the wire at points about half a millimetre apart, as shown in Fig 88. The electro-magnet is mounted on a small base-board. As a refinement, adjusting screws may be fitted, bearing against the sides of the magnet so that the amount of "stagger" of the pole tips may be varied.

For the amplifier, two-valves transformer coupled will suffice, but a three-valve circuit is better. For the output stage, a pentode is advisable. A step-down transformer is used with a change-over switch so that its secondary may be connected to the recorder or loud-speaker. A small moving-coil loud-speaker makes an excellent microphone. Theappings giving the largest step-up ratio are connected to the input terminals of the amplifier. When playing back the record, the coils C C may be connected to the input terminals through the same transformer, another change-over switch being used.

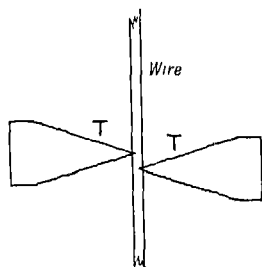


FIG. 88

To prepare the wire for recording, hold a horse-shoe magnet against it for one circuit. Connect the microphone to the input, and the output to the coils C C. Speak a few words into the microphone. Switch over input transformer to coils C C and output to loudspeaker. Sounds bearing some resemblance to the original words should then be heard from the loudspeaker at every circuit of the wire. To remove the record, magnetize the tape with a horse-shoe magnet as before.

Increasing the distance between the pole tips leads to loss of high frequencies; reducing this distance causes loss of bass.

## 186. PHOTO-ELECTRIC SIREN

*J. P. Stephenson*

In the ordinary disc siren, sound is produced by interrupting a blast of air by holes in a circular disc. The same sound effects can be produced by interrupting, at regular intervals, a beam of light which is falling on a photo-electric cell. The resulting pulses of current are passed through an amplifier and transformed into sound by a loudspeaker. If a siren disc is not available, one can be made for a few pence from a disc of aluminium

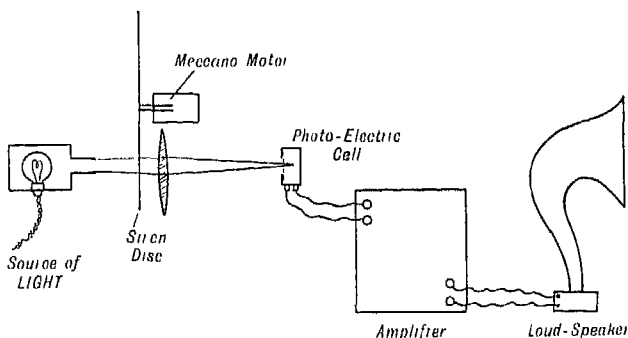


FIG. 89.

and fitted to a "Meccano" motor. When the beam passes through the row of regular holes on the inner circle, a musical note cannot be obtained, but the other notes give *doh, me, soh, doh*. A tuning-fork, fitted with a slit, will of course serve the same purpose, but more light gets through the siren disc.

A Weston "Photronic" cell has a good output and the amplification stages of an old portable wireless will give sufficient volume. This experiment also serves to show the principles involved in the production of sound from film.



## 187. LEMON CELL

*E. J. Harris*

This experiment demonstrates the fact that any two dissimilar conductors, placed in an electrolyte, constitute a voltaic cell. A knife and fork are stuck into a lemon and connected to a sensitive galvanometer or millivoltmeter. A considerable deflection is shown. (If the fork is replaced by a piece of copper, a sufficient voltage is produced to give a measurable deflection on an ordinary voltmeter.)

## 188 POLARITY OF MAINS

*H. C. Palmer*

Cut a potato in halves. Thrust the two wires into the freshly cut surface. The potato around the anode is stained blue. A lamp in series is a wise precaution.

## 189. HOME-MADE ELECTRIC SIGN

*R. R. Finney*

This consists of four parts : (1) the motor, (2) connection with rotating arms by means of a gear, (3) the fibre square with the studs, over which rotates the arm, (4) the electrical connections.

*The Motor.*—This is a second-hand electric gramophone motor obtained through "Exchange and Mart" for 10s. A wooden board, 18 in.  $\times$  9 in.  $\times$   $\frac{1}{2}$ -in., forms a convenient and substantial base on which to fix the parts. The motor is fixed on end near one end of the board by means of Meccano angle irons, so that the rotating spindle is  $5\frac{1}{2}$  in. above the board and facing so that it can engage the geared connections. Through the end of the spindle is passed a split pin, which engages a split cup connected

to the train of gears. This arrangement gives a perfectly satisfactory drive

*Gearred Connections.*—These were obtained from a milk separator, a local engineer making an iron stand by which they were firmly fixed to the base-board by means of nuts and bolts.

*Fibre Distribution Board, etc*—This is 9 in. square by  $\frac{1}{2}$ -in. thick and fastened by screws through the base-board and also to the cover of the gears, fibre cylinders giving rigidity in the latter case. Through the centre of the fibre board passes the end spindle of the train of gears

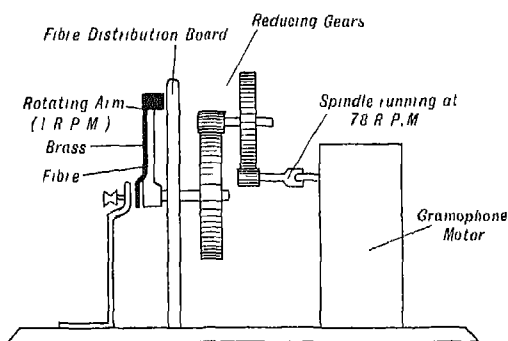


FIG. 90.

and is fixed to the rotating arm, which moves over the brass studs. Forty of these are placed at equal distances apart on the circumference of a circle of 4 in. radius. The flat ends of the studs are brought flush with the surface of the fibre board on the rotating arm side. The other ends of the studs are threaded and a small nut fixed to facilitate wire connections. The flat end of the studs is circular, of about  $\frac{1}{4}$ -in. diameter.

*Rotating Arm* (see Fig 91).—This is a piece of fibre, 4 in.  $\times$   $\frac{1}{2}$ -in.  $\times$   $\frac{1}{4}$ -in., fixed to the spindle, which projects  $\frac{1}{2}$ -in. through the fibre distribution board. The fibre arm is attached to the spindle by a brass U, the arms of which are  $\frac{3}{4}$ -in. long. A screw through the ends of the arms of the U and the fibre arm makes a firm connection. The

outside of the rotating arm is covered with brass strip, which is fixed to the fibre by small nails. The stud end of the strip has a small brass cylinder attached to it and is long enough to slide smoothly over the studs and to make good contact with each. The other end of the brass strip bends away, about  $\frac{1}{2}$ -in from the fibre arm,

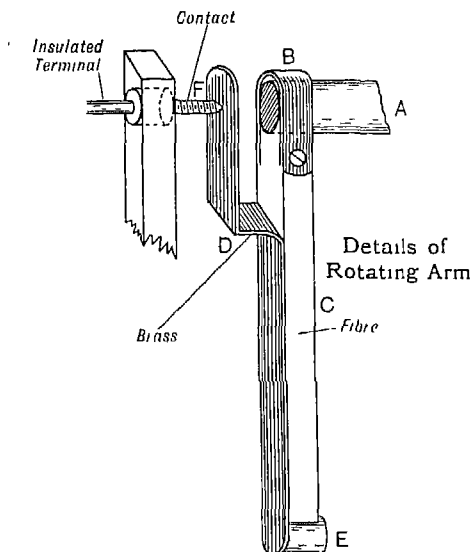


FIG. 91.

- A. Spindle through fibre board from gears.
- B. Brass U, joining arm to A
- C. Fibre arm
- D. Brass strip, fastened to C by nails.
- E. Cylinder, soldered to D and making contact with studs.
- F. Screw closely touching depression near end of D.

and a slight depression at the end on the outer surface makes contact with the screw end of the terminal, which is passed through a hole in the top of the long arm of a brass angle piece,  $4\frac{1}{2}$  in.  $\times$   $1\frac{3}{4}$  in.  $\times$  1 in. wide. Two fibre washers insulate the terminal from the angle piece. The shorter arm of the brass angle piece is screwed to the wooden base.

*Electrical Connections.*—Numbers 1–40 represent studs

on fibre board. Letters A-G represent lamps (seven shown). O represents moving arm, rotating about centre L. The connections between lamps and studs are behind the fibre board, as well as cross-connections between the studs.

To maintain the lamps flashing regularly, cross-connections are made between the studs. In the case under consideration (seven lamps), No. 2 stud is connected to No. 10 and then to No. 18, No. 26 and No. 34, the wavy

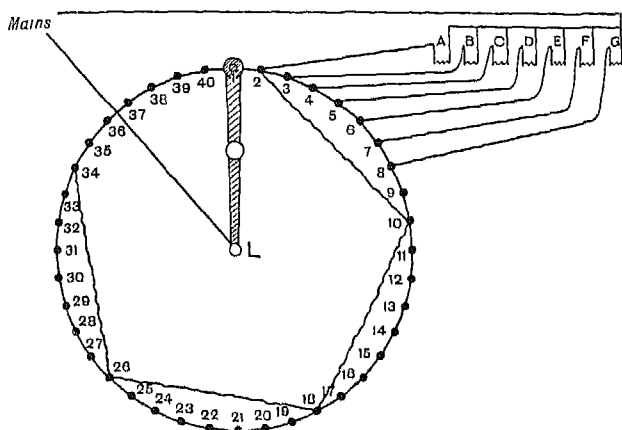


FIG. 92.—Plan of Rotating Arm (full size).

lines show the connections. Similarly, studs Nos. 3, 11, 19, 27 and 35 are connected, and so on. Thus, when the arm rotates over studs Nos. 2, 10, 18, 26 and 34, lamp A will light. Studs 9, 17, 25, 33 and 1 are blanks. Other numbers of lamps, up to 40, lend themselves to various arrangements.

*All connections are made with rubber-covered wire.*

## 190. "IONIC ENGINE"

*W. Bryan Chivers*

This is an interesting piece of apparatus to demonstrate the ionization of the air by a flame. A light wooden

beam, about 8 in. in length, is supported on two gramophone needles (Fig. 93), a plate of brass being fixed in the centre with punch-holes to locate the needles. By means of a lead wire rider, the beam can be brought into delicate equilibrium. The beam carries at one end a brass plate, its edges carefully smoothed, and at the other a copper contact piece. Another brass plate is held by a plug of sulphur-wax just above the movable plate. The flame of the night-light is in contact with this plate. The beam normally rests on the stop, but when sufficient charge has been collected by the candle, the upper disc attracts the lower one, which rises until it touches and discharges the

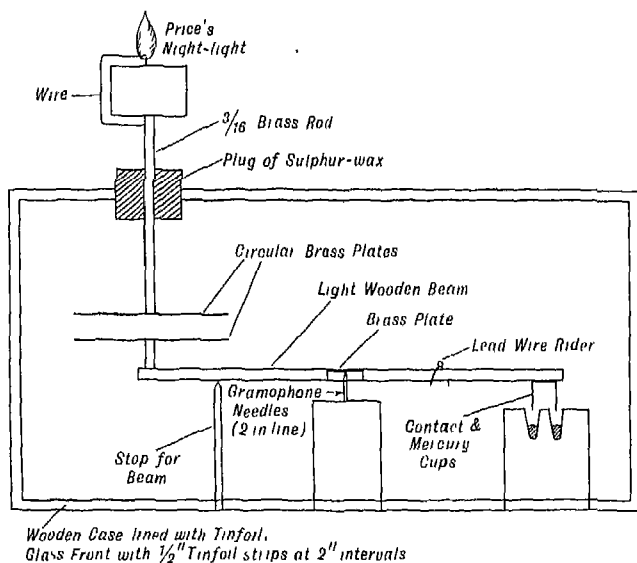


FIG. 93.

upper one, the conductivity of the wooden beam being sufficient to allow the charge to escape the earth. At the rising of the beam, the mercury contacts close and a lamp may be lit intermittently. The case is best made of metal, but wood can be used with glass side if the wood is covered

with tinfoil, and pieces of tinfoil attached at intervals along the glass plate.

The machine that I made tilted its beam every fifteen seconds and went on working for hours at a *conversazione*, to the great mystification of the beholders.

## 191. JUMPING BALL

*J. P. Stephenson*

This is a modification of the well-known experiment in which a soft iron bar is drawn into a coil carrying a current. A reel of 26 gauge d.c.c. wire (7 or 8 lb.) stands on a tripod; a bar of soft iron rests on the bench in line with the axis of the coil. Since the self-induction of the coil is large, the ends can safely be joined to 220 v. mains, and the bar is drawn up. When it reaches the top of the reel, it strikes a brass ball. This ball rests on two pieces of Meccano strip, fixed to the inside of a funnel; one of these strips is connected directly to the funnel; the other is insulated.

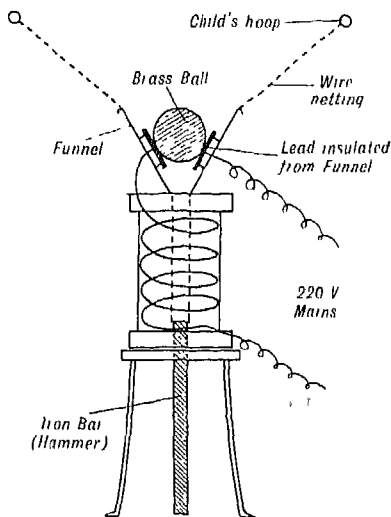


FIG 94.

It will be seen from the diagram that the ball is made to complete the mains circuit, so that when it is projected upwards by the iron bar, the current is stopped and the bar falls down. When the ball drops back into the funnel, the whole process is repeated. A ball of about  $1\frac{1}{2}$  in. diameter can easily be projected about 10 ft.

The ball does not normally fall back into a 4-in. funnel,

but a child's hoop, of about 18 in. diameter, can be used to make a larger catchment of wire netting. Various other modifications can be introduced to make the apparatus quiet in action. A one-microfarad condenser across the break will quench the rather big spark. The bar can be fitted with a rubber tip (small bung), and a pad of sorbo on the bench stops the banging. In place of the brass ball, which may be thought dangerous, a fives ball, wrapped with a mesh of bare copper wire to make it conducting, can be used.

## 192. JACOB'S LADDER

*J. P. Stephenson*

Two pieces of stout copper wire, about a yard long, are held vertical and nearly parallel by threads attached to a wooden frame. If

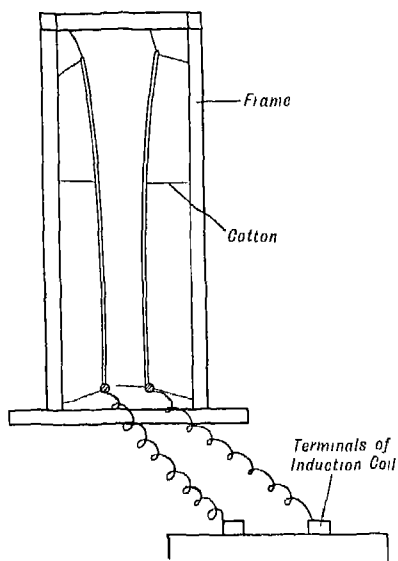


FIG. 95.

the wires are about 3 in. apart at the bottom and 4 in. at the top, they serve as sides to the ladder, and when joined to the secondary of a fairly large induction coil, a spark beginning at the bottom will gradually rise up the ladder. The experiment can be made most spectacular if carried out in a dimly-lighted room, and if the wire has a coating of strontium, barium and other salts, so that the "rungs" of the

ladder then change colour on the way up

## 193. GLOW-TUBE OSCILLOGRAPH

*J. P. Stephenson*

A fluid-light visual-tuning indicator can be used as an oscillograph. The voltage required to "flash" these lamps depends on the type, but the "Cossor" bulb will flash on 160 volts. The whole cathode is covered with glow at 170 volts. These bulbs generally have three electrodes—two small ones to strike the tube and the cathode to which the variable voltage is applied. The diagram gives full details of the circuit, but the values

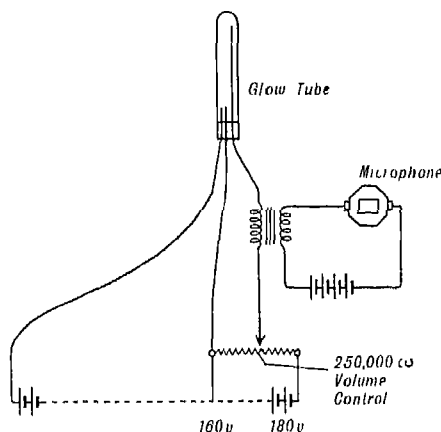


FIG. 96.

for the transformer will depend on the microphone. For examining speech currents, the output of a microphone is applied to the cathode through a transformer (bell-ringing type). The glow will dance up and down the tube, and on viewing in a rotating mirror, the form of the currents can be seen. The discharge curve for a small condenser can be observed in a similar way. Some tubes give a better "top" to the glow than others and thus clearer patterns. It is a matter of luck, for the tubes are not intended for this purpose. The batteries used are 120 and 60 v. H.T., connected in series.



## 194 FLASHING LAMP

*J. P. Stephenson*

The ordinary fairy lights and many sign devices depend on the bimetallic strip for breaking the circuit. In the Christmas fairy lights, the bimetallic strip is included in one of the lamps, called a "flasher." If the spray is washed off this bulb by acetone, it will be seen that the strip receives heat from a filament, and the consequent bending breaks the circuit. Since all this takes place in the vacuum, there is no appreciable spark.

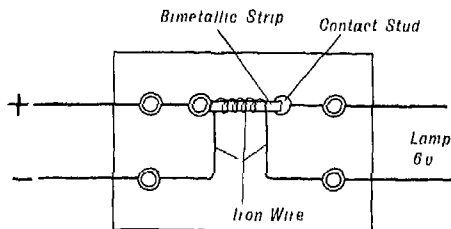


FIG. 97.

This strip, made of invar and brass welded together and then rolled out, can be obtained from an instrument-maker or metal shop. A strip,  $2\frac{1}{2}$  in. long and  $\frac{1}{4}$  in. wide, can be used to make a model flasher. A hole is drilled in one end to fit a terminal; the other end can rest on a contact stud. Gummed paper should be bound round the strip as insulator, and about a yard of iron wire makes a suitable heater (if a 6-v. lamp is used); this should be wound round the metallic strip over the paper.

From the diagram it will be seen that the current flows until the heater bends the strip and thus breaks the contact.

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